The Effects of PET-Lined and Epoxy-Coated Lead and Copper Service Lines on Metals Leaching, Total Organic Carbon, and Chlorine Residual in Drinking Water

by

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ABSTRACT

Lead service lines (LSLs) can represent a significant health and economic issue for utilities that find elevated lead levels in tap water samples collected under the requirements of the Lead and Copper Rule (LCR). Full LSL replacement can be expensive if trenching is required. Additionally, the LCR does not require replacement of the homeowner-owned portion of the LSL. When utilities replace only their portion (partial replacement), the remaining disturbed LSL can contribute significant spikes in lead levels and continuing lead release. Application of a lining or coating may provide a more effective and less expensive alternative than partial LSL replacement, both for reducing corrosion and for increasing homeowner participation. However, it would be wise to first evaluate the impact the product itself will have on water quality through chemical leaching and chlorine consumption.

Fill-and-dump experiments were performed to determine the impact of a commercially available epoxy coating and poly(ethylene terephthalate) (PET) liner on water quality in lead and copper pipes. Forty-five-inch-long pipe sections, including both LSL sections obtained from Rochester, N.Y. and sections of commercially available soft (annealed) Type L copper tubing were either coated with epoxy or lined with PET. These pipe sections were then tested using three types of extraction water: dechlorinated tap water and chlorinated extraction water (both at pH 8), and extraction water at pH 6.5. The chlorinated pH 8 extraction water and the pH 6.5 extraction water were prepared by dosing reagent water with NaCl, $CaCl_2$, and $NaHCO_3$, and were similar to waters outlined in NSF International 61 testing protocols. Holding times were varied to represent a range of detention times experienced by LSLs in use. Unlined lead and copper pipe sections were used as controls. In addition to lead and copper, the extraction waters were analyzed for chlorine demand, total organic carbon (TOC) concentration, and also for antimony in samples exposed to PET liner, which was found to contain ~130 mg Sb/kg PET. Partially digested epoxy was found to contain 8 mg Cu/kg.

Extraction water exposed to the uncoated and unlined (control) lead and copper pipes contained 1,200 – 25,000 μg/L lead, and 270 – 910 μg/L copper, respectively. Extraction water exposed to epoxy-coated pipes had significantly less lead, with most samples below the detection level of 0.5 μg/L. Only one pipe specimen yielded a lead concentration above the action limit (AL) of 15 μg/L, and two subsequent extractions of the same pipe specimen yielded lead levels below the detection limit. All extraction waters exposed to epoxy-coated lead and copper pipes contained increased levels of copper (0.4 – 22 μg/L), although significantly less than those from the uncoated control and far below the AL of 1300 μg/L. This may be due to leaching of trace amounts of copper from the epoxy itself. Epoxy-coated pipes exerted significant free and combined chlorine demand, with nearly all chlorine consumed after 6 hours when exposed to an initial free chlorine or monochloramine concentration of 2 mg/L as Cl_2, a demand similar to that of the uncoated control specimens. After repeated exposure, the free chlorine demand stabilized at approximately 10% of the initial free chlorine concentration in pipe specimens exposed to either 2 mg/L as Cl_2 for 1 hour or 100 mg/L as Cl_2 for 3 hours. Pipe specimens freshly coated with epoxy leached a statistically significant amount of TOC (an average of 0.65 mg/L) into the chlorinated pH 8 and pH 6.5 extraction waters, but there was, on average, no measureable increase in TOC in samples exposed to dechlorinated tap water.

A statistically significant increase in antimony concentration was observed over time in PET-lined pipe specimens. Antimony concentrations in samples of extraction waters exposed to PET-lined specimens increased by an average of 0.09 and 0.33 μg/L in specimens exposed for 6 – 24 hours and 4 days, respectively, but the antimony increases remained more than an order of magnitude below the maximum contaminant level (MCL) of 6 μg/L. Free chlorine levels decreased only by half over 4 days when PET-lined pipes were exposed to an initial concentration of 2 mg/L as Cl_2. When the same PET-lined pipes were again exposed to free chlorine for two or three 24-hour periods, the 24-hour chlorine demand was only about 5 percent of the initial chlorine concentration, approximately the same as in control samples of the chlorinated pH 8 extraction water. Thus, the PET liner exhibited no significant long-term chlorine demand. Lead and copper data collected from PET-lined pipe specimens were compromised by use of incompatible end fittings that failed to seal water flow during flushing, highlighting the importance of proper preparation of pipes for effective lining or coating installation. PET-lined pipe specimens leached an average of 0.15 mg/L TOC into chlorinated pH 8 extraction water. This small but statistically significant increase may have been associated with sample handling or small variations in instrument response rather than leaching, and no statistically significant increase in TOC was observed using the other extraction waters, including dechlorinated tap water.

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CHAPTER 1: INTRODUCTION

Lead service lines (LSLs) can represent a significant health and economic issue for utilities that find elevated lead levels in tap water samples collected under the requirements of the Lead and Copper Rule (LCR). When lead levels are measured above 15 μg/L in more than 10% of the samples, the LCR requires utilities to take action (EPA 2013). Since LSLs can be a significant contributor to elevated lead levels measured in tap water samples, replacement of LSLs is a required action for utilities if the action level is still exceeded after implementation of corrosion control processes (Sandvig *et al.* 2008). However, replacement of the homeowner-owned portion of the LSL is not required under the LCR. When utilities replace only their portion (partial replacement), the remaining disturbed LSL can contribute significant spikes in lead levels and continuing lead release (Griffiths *et al.* 2011). When copper pipe is used to replace a portion of the LSL, lead release can be accelerated in some cases due the formation of a galvanic cell (Triantafyllidou and Edwards 2011). Furthermore, full LSL replacement can be expensive if trenching is required. Utilities and homeowners could face widely varying costs of up to $3600 to replace their portion of the LSL (Boyd *et al.* 2000).

Application of a lining or coating may provide a more effective and less expensive alternative to partial LSL replacement, both for reducing corrosion and for increasing homeowner participation. Typical lining and coating installations require pits dug at only the ends of the pipe, saving intensive labor and machinery costs associated with trenching along an entire pipe. Fewer trenches could lead to less land disruption, fewer traffic delays, and a smaller construction site footprint. Homeowners could realize and appreciate similar benefits and be more inclined to participate due to less destruction to their property by avoiding trenching.

However, before installing a lining or coating, it would be wise to first evaluate the impact it may have on water quality through chemical leaching. Studies of plastics similar to those used for linings have found leaching of metals such as antimony (Cheng *et al.* 2010; Peric-Grujic *et al.* 2010; Reimann, Birke, and Filzmoser 2010; Shotyk and Krachler 2007). The leaching of organic chemicals, such as bisphenols and phthalates, that are ubiquitous in plastics and epoxies, respectively, is also of concern due to their potential as endocrine disrupting compounds (EDCs) and possible role in forming disinfection by-products (DBPs). A companion study (Lane *et al.* 2013) is focused on the aspect of organic chemical leaching in linings and coatings. In this study, fill-and-dump tests were performed to determine the effectiveness of commercially available linings and coatings at preventing release of lead and copper into drinking water and to determine whether the linings and coatings themselves consume chlorine or leach metals or total organic carbon (TOC) into drinking water.

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CHAPTER 2: BACKGROUND

LEAD SERVICE LINE HISTORY AND USAGE

Although lead water pipes were a public concern as early as the 1850s (Kirkwood 1859), large scale installation began in the late 1800s in the more populous United States cities (Rabin 2008). Being more corrosion resistant and malleable than readily available cast iron, lead pipes lasted longer and allowed installers to easily bend pipe around existing obstacles. Installation of lead water pipes began to diminish in the 1920s due to increasing public concern, but remained significant through the 1950s and was not banned outright until the Safe Drinking Water Act Amendments of 1986 (Rabin 2008). The actual number of lead service lines remaining in service in the United States today is unknown. However, 51 utilities who responded to a 2007 survey estimated 1.1 million lead service lines were still in service in their distribution systems, or 12% of these utilities' service lines (Sandvig *et al.* 2008). In the same 2007 survey, an additional 368,000 lead connections, including goosenecks, were reported from 31 utilities.

HEALTH ISSUES ASSOCIATED WITH LEAD

Exposure to lead causes adverse health effects, and has been widely studied. Children are most affected by lead exposure, as blood lead levels (BLL) of 15 – 30 micrograms per deciliter (μg/dL) can cause neurological damage such as diminished psychomotor performance, reaction time, and intelligence quotient (IQ). Encephalopathy, or brain disease, and even death can occur in children with a BLL of 80-100 μg/dL (EPA 2006). Renewed calls for lowering the allowable BLL have cited recent research that indicates neurological damage at BLLs below 10 μg/dL, including a decrease of one IQ point at a BLL as low as 0.1 – 1.0 μg/dL (Budtz-Jergensen *et al.* 2013). If the health effects from lead poisoning are irreversible, exposed individuals have to live with the inflicted damage for the rest of their lives. From a public health perspective, if the damage cannot be treated, then it must be prevented.

THE LEAD AND COPPER RULE (LCR)

In an effort to decrease lead consumption by the public from drinking water, the United States Environmental Protection Agency (EPA) published the Lead and Copper Rule (LCR) in 1991 (EPA 2013). The LCR requires water utilities to test samples taken from households considered to be at high risk for high lead levels according to a defined tier system. The number of sites to be sampled and frequency of sampling is dependent on the number of consumers served but can be reduced if very strict criteria for water quality or very low lead levels are demonstrated and maintained. Utilities must take corrective action if more than 10% of samples contain ≥ 15 μg/L lead, referred to as the AL. For comparison, the AL for copper is 1300 μg/L. Actions that can be taken by utilities to correct high lead levels include optimized corrosion control treatment (OCCT) and source water monitoring and treatment. If utilities continue to exceed the AL after implementing OCCT and/or source water treatment, they are required to replace at least 7% of lead service lines (LSLs) (based on the initial number of LSLs at the time the replacement program begins) per year. Replacement of LSLs can be discontinued when ALs are not exceeded for 2 consecutive 6-month monitoring periods, but must be re-continued if the

AL is exceeded again. A key option for utilities is the ability to consider individual LSLs as "replaced through testing" if all samples collected from the LSL are below the AL. However, these LSLs must be reconsidered if later sampling results exceed the AL.

Sampling procedures for monitoring lead and copper for LCR compliance are outlined by EPA (2013). One sampling method is referred to as 'first draw'. One liter of cold tap water is taken from a household's fixture from which water is consumed, typically the kitchen sink. The sample must have been allowed to stagnate in the premise plumbing for at least 6 hours. For the purpose of determining if an individual LSL requires replacement, sample collection is designed to obtain water that was allowed to stagnate in the LSL for at least 6 hours. Instead of collecting the first draw, the calculated volume of water between the tap and LSL is flushed prior to sample collection. The collector can also flush until a significant change in water temperature is observed or, less commonly, tap into the LSL itself to collect a sample. After collection, samples are acidified with nitric acid for preservation.

LEAD SCALES

Solid lead exposed to the constituents found in typical drinking water can create a number of different lead sequestering scales, primarily in the form of lead-oxides and lead-carbonates. Other scales, such as calcium carbonate, can also form on drinking water pipe walls. When scales are stable they can physically block further lead release from the inner pipe wall. Furthermore, dissolved lead can be removed from drinking water by integration into a forming scale. However, scale stability depends on a number of factors, including redox potential, dissolved lead concentration, carbonate concentration, pH, hardness, and others that all vary in different drinking waters. Strategies for OCCT programs implemented for LCR compliance include optimizing water quality parameters such as pH, carbonate, or phosphate levels in order to encourage scale formation or to retard dissolution of lead or lead-containing scales. The optimal program depends on each drinking water's unique water quality (Schock 1989; Spencer 2003).

Phosphate addition at the treatment plant has been shown to be particularly effective at reducing lead levels in drinking water. Orthophosphate has the ability to interact directly with the interior surface of the lead pipe wall while polyphosphates combine with calcium to form a protective scale barrier (Boffardi and Sherbondy 1991). However, these scales never reach equilibrium in a water pipe because their physical and chemical stability is fragile and can be disturbed by changes in water quality or flow. For instance, recent research indicates lead dioxide is stable in drinking water chlorinated with sufficiently high concentrations of free chlorine, which drives the oxidation of lead to lead(IV). A switch to combined chlorine, or lower levels of free chlorine, changes the oxidation reduction potential of the water and lead(IV) is reduced to lead(II), destabilizing the lead scale (Boyd *et al.* 2008; Lytle and Schock 2005).

As drinking water moves through the distribution system, its pH can vary significantly from what was measured at the treatment plant (Spencer 2003). Consequently, the stability of scale formation can vary from location to location in the distribution system. For instance, a relatively narrow pH range of 7.2 -7.8 is recommended for orthophosphate addition to be effective. Lead service lines, a major potential source of elevated lead levels, are located at the

end points of a distribution system. Drinking water could therefore undergo a significant pH change by the time it reaches the component OCCT is meant to target. A more universal method to control lead levels, one that can remain effective through broad water quality ranges, could be beneficial to utilities that find OCCT either ineffective or prohibitively expensive for their system.

LSL CONTRIBUTION TO ELEVATED LEAD LEVELS

Sandvig et al. (2008) conducted a study on historical data and their own sequentially collected tap samples to determine lead contributions from the faucet, premise plumbing, LSL, and meter. They concluded that 50 – 75% of the lead originates from LSLs. The first flush samples could therefore be an inadequate representation of the risk of a site's potential for elevated lead levels since they only include water stagnating in premise plumbing.

The amount of lead that reaches the tap depends on a number of factors, including LSL length and diameter as well as drinking water qualities that affect lead corrosion. As LSL length increases and diameter decreases, the lead surface to water volume ratio increases and, therefore, the potential for lead leaching also increases. At redox potentials typically seen in drinking water, solid lead oxidizes to Pb(II) (its most environmentally common oxidation state) more readily as pH decreases (Vasquez *et al.* 2006). When partial LSL replacement is performed, the lead portion remaining has been shown to continue releasing lead (Boyd *et al.* 2004). When copper is installed in place of the portion removed, galvanic corrosion can accelerate lead release in some cases (Cartier *et al.* 2013; Triantafyllidou and Edwards 2011). Half of the 22 utilities who answered the 2007 EPA survey described their LSL replacement as partial, 5 of which typically left more than half of the existing LSL in place (Sandvig *et al.* 2008).

Lead-bearing particles that break off intermittently and inconsistently into the drinking water supply are far less likely to be captured by periodically collected first-draw samples under the LCR than the dissolved lead that results from general corrosion. When particulate lead is captured, significant lead spikes can occur in samples. Increased particulate lead levels can result from higher flow rate that can destabilize accumulated lead scale layers or suspend heavier solids (Cartier *et al.* 2013). Some investigators have seen spikes above 1000 µg Pb/L, or 67 times the AL, immediately after partial LSL replacement, due mostly to particulate lead (Boyd *et al.* 2004; Sandvig *et al.* 2008). Solder used in the service line can also play a crucial role in spikes of particulate lead as the solder ages, deteriorates, and breaks off. Disturbing the pipe, as in the case of LSL replacement, increases the risk of the particulate lead reaching the tap. In a study of Washington, D.C. drinking water, particulate lead only comprised approximately 10% of the lead in samples collected for LCR compliance (EPA 2007). However, the amount of lead measured may not reveal the total lead reaching the tap due to its dependence on sampling and measuring methods. Inspected faucet aerator screens taken from the apartment building of a lead poisoned child in North Carolina revealed trapped particles of lead solder. These solids, along with lead(IV) oxides, may not dissolve completely in some cases with current EPA methods used to test samples collected under the LCR (Triantafyllidou, Parks, and Edwards 2007). The lead measured can also vary due to the length of stagnation time (at least 6 hours under the LCR) and flushing time used prior to sampling.

COST CONSIDERATIONS

Although an old LSL can be pulled from the ground without excavating, a new trench is often required for its replacement, especially in situations where a pipe could potentially damage nearby utility lines or structures if pulled. Trenching represents a significant portion of service line installation costs due to the machinery and labor involved. Furthermore, if a newly located trench is required, conflicts with other utilities can arise. In the 2007 survey performed by the EPA, 80% of the responding utilities said that customers were responsible for all aspects of replacing their portion of the LSL (Sandvig *et al.* 2008). Despite the health risks, relatively few homeowners opt to replace their portion of the LSL. Cost may be a major factor in homeowner's decision making. A 2006 survey conducted of utilities lists the cost of replacing the utility owned portion of a LSL at $250 – 3,000 and the homeowner's portion at $600 – 4,000 (Sandvig *et al.* 2008). A survey conducted by Boyd *et al*. in 2000 lists the cost to utilities for open trench replacement of LSL at $240 – 3,600. Slip lining can cost $450 – 700 and pipe coating $900 – 1,100. A significant portion of the cost savings comes from the lack of excavating an entire trench for lining and coating installations. Instead, a pit is typically dug at each end of the pipe in order to connect the installation equipment. The lack of trenching could also minimize traffic delays and destruction to the land surface which, combined with the cost savings, could help entice homeowners to participate in replacing their own portion of the LSL.

LININGS AND COATINGS AS AN ALTERNATIVE TO LSL REPLACEMENT

Clearly, LSLs represent a significant health and economic issue when they continually release lead above the AL of 15 μg/L and require replacement. In some cases, the common practice of partial replacement may not be an effective method to reduce lead levels. Full or partial replacement can also be cost prohibitive to utilities that need to replace multiple LSLs. Of the 28 utilities who responded to the 2007 EPA survey question of whether alternative treatments were considered to manage lead levels at the tap, 23 had not considered these options (Sandvig *et al.* 2008). Linings and coatings provide a means for utilities and homeowners to cut costs associated with trenching, and may offer a more effective alternative to blocking continuing lead release from partial LSL replacement. Furthermore, the physical blocking of lead release from the inner pipe wall would not be dependent on water quality as in the case of scale formation.

Linings and especially coatings can conceivably be carried into the premise plumbing in some cases. Over the course of decades in which household plumbing is serviced by a LSL, released lead has the potential to 'seed' the plumbing by forming scales or getting trapped in tight bends in the case of larger lead particles (Deshommes *et al.* 2010). If the seeded plumbing remains after LSL replacement, elevated lead levels could continue to reach the tap. Other premise plumbing issues such as pinhole leaks in copper pipes, or excessive corrosion such as pitting, can benefit from lining and coating technologies. Furthermore, copper service lines (CSLs) are used extensively by water utilities; thus, there is also interest in technologies that can be used to rehabilitate CSLs, although violations of AL for copper are rare and do not result in required replacement.

LINING AND COATING INSTALLATION

Before a lining or coating can be installed, the pipe must be prepared. For epoxy coatings, the pipe is typically sandblasted to remove excess scale and provide a rough surface that improves the adherence of the epoxy to the inner pipe wall. Flushing is then performed with water to remove debris, and the pipe is dried. Epoxy can then be blown into the pipe by applying air to control pressure, which has the effect of creating a vortex through the pipe and spreading the epoxy evenly along the pipe. After the coating is finished, airflow is usually continued to facilitate curing and minimize pooling. Curing time can range widely depending on the proprietary mixture, of which there are many currently in the marketplace or under development. After curing, the pipes are flushed and can be put back into service.

Before installing a lining, the pipe is typically scraped or pigged. Sandblasting is not required since the liner does not need to adhere to the inner pipe wall. Scraping or pigging is performed to restore water flow capacity and ensure no sharp obtrusions remain to compromise the integrity of the liner. The process of installing a poly(ethylene terephthalate) (PET) liner involves inserting PET tubing of smaller diameter than the pipe being lined. Hot water is pumped under pressure to force the liner to expand and fit tightly onto the inner wall of the pipe. The liner is then flushed and can be quickly put back into service due to the lack of need for a curing time.

CONCERNS ABOUT LININGS AND COATINGS

The type of plastic used in linings can vary, but many consist of some form of PET, as was the lining tested in this study. Antimony, in the form of Sb_2O_3, is used in 90% of the world's PET production (Hansen and Pergantis 2006). In most applications, 190 – 300 parts per million (ppm) antimony is used to produce PET (Duh 2002). The EPA has established a maximum contaminant level (MCL) of 6 μg/L for antimony due to its harmful toxicological effects. Many have studied the leaching of antimony into bottled water (commonly made of PET) and have found levels of 0.1 – 1.8 μg/L with some results approaching the MCL when the PET is exposed to high temperatures, as experienced if stored in a vehicle during summertime (Cheng *et al.* 2010; Peric-Grujic *et al.* 2010; Shotyk and Krachler 2007). Although antimony release is seen to increase with increasing temperature and storage time, it is independent of pH (Cheng *et al.* 2010; Reimann, Birke, and Filzmoser 2010). An advantage of using plastic linings or coatings in service lines is their burial shields them from extreme temperatures and temperature fluctuations that could accelerate leaching and fatigue. Plastic water bottles would theoretically experience longer detention times and higher temperatures than experienced in a service line and therefore could be assumed to represent a worst-case scenario for PET-lined LSLs. However, since some studies show antimony levels in water bottles approaching the MCL, it would be prudent to test LSL lining products under conditions similar to those experienced in practice.

Identifying inorganic compounds in epoxies is difficult since there is no typical epoxy used in all coating installations. Epoxies are made from a myriad of different formulations, each tailored to its intended application. Additives can be included to change a number of characteristics, including curing time, color, and flexibility. A clear picture of inorganic

constituents in epoxies would require a comprehensive study on numerous different formulations, and is not in the scope of the research presented here. Some epoxies are formulated for potable water applications, including the epoxy tested in this study. Epoxies used specifically for coating drinking water pipes are assumed to have similar abilities to block lead release. As with plastic liners, epoxies used in drinking water applications should be tested for leaching under conditions experienced by service lines. One form of leaching in amine-based epoxies, referred to as 'blooming,' occurs in instances where curing has been interrupted. Incomplete curing leads to a lower degree of crosslinking, and can happen when amine concentrations are substantially greater than stoichiometrically required (Petrie 2006; Sherwin Williams 2008). When leaching amines reach the surface, they react with carbon dioxide to form ammonium carbamate. Long-term effects of leaching include epoxy discoloration and delamination from the substrate (Dow Chemical Company 2007) or, in the case of a coated service line, the pipe wall.

The effect of linings and coatings on chlorine demand is another concern. Chlorine residuals in the distribution system are meant to discourage biological growths that can lead to bacterial infection as well as taste and odor issues. If a lining or coating exerts a chlorine demand, the potential for biological growth could rise. Pipe-wall demand occurs when chlorine reacts with the inner pipe wall or anything that forms on its surface, such as biofilm or tubercles. Important factors that influence wall demand include: initial chlorine concentration, corrosion, biofilm, pipe material, diameter, and age. Pipes made from metals such as cast iron, steel, and copper tend to react with chlorine more than synthetic materials such as polyvinyl chloride (PVC), high-density polyethylene (HDPE), and medium-density polyethylene (MDPE). Furthermore, chlorine demand in metal pipes can increase with age in some cases (Al-Jasser 2007; Hallam *et al.* 2002; Kiene, Lu, and Levi 1998).

In 72 – 96 hour fill-and-dump experiments, epoxy-lined copper pipes have been observed to consume approximately 1.5 mg/L free chlorine as Cl_2 or 1 mg/L monochloramine as Cl_2 when initial test waters contained 2 mg/L free chlorine as Cl_2 or 4 mg/L monochloramine as Cl_2, respectively (Heim and Dietrich 2007). Chlorine could be consumed by the chlorination of polyamides, which has been proposed to occur in both reversible and irreversible reactions (Kawaguchi and Tamura 1984) that lead to cleavage of the interchain crosslinks and polymer chain (Koo, Petersen, and Cadotte 1986; Singh 1994). The chlorine demand exerted by epoxy could potentially diminish over time after repeated exposure to chlorine if the reaction occurs at the surface. However, chlorine demand could continue if chlorine diffuses into the epoxy or amines leach out. Therefore, a study to determine the long-term effects epoxy has on chlorine demand is needed.

The leaching of organic chemicals that are ubiquitous in plastics and epoxies, such as phthalates and bisphenols, respectively, is also of concern due to their potential as endocrine disrupting compounds (EDCs) and role in forming disinfection by-products (DBPs). If organic chemicals are observed to leach from epoxy coatings or PET liners, and if a significant chlorine demand is also observed, there could be significant potential for DBP formation. Other organic compounds, such as humic and fulvic acids, have the potential to complex with dissolved metal ions such as iron and copper. Quantifying the amount and type of organic chemicals, such as

bisphenols and phthalates, leaching from linings and coatings is beyond the scope of this study, but is being performed in a companion study (Lane *et al.* 2013).

As mentioned earlier, the LCR already offers a provision for utilities to forego individual LSL replacement if they can demonstrate lead levels below the AL in all samples taken from that LSL (CFR Title 40 Part 141.84(c)). Utilities could have a less expensive alternative to consider when addressing LCR compliance if the LCR can be amended to include foregoing individual LSL replacement after rehabilitation via lining or coating and/or demonstration of lead levels below the AL after rehabilitation. Furthermore, households not constrained by the LCR could take action themselves by utilizing a lining and coating technology to rehabilitate their portion of the LSL and/or premise plumbing. To facilitate wise decisions by lawmakers or regulatory agencies considering changes to the LCR and utilities and homeowners considering linings and coatings as a viable alternative, the parties involved need information regarding the effectiveness of lining and coating technologies to block lead release and their susceptibility to leach chemicals into drinking water.

OBJECTIVES

The objectives of this research are to determine the effectiveness of linings and coatings to block the release of lead and copper, their potential to leach constituent metals, and their effect on TOC and chlorine concentrations in drinking water. To investigate these issues, fill-and-dump experiments were performed on lead and copper pipe sections, either lined with PET or coated with epoxy, as well as unlined lead and copper pipe sections. Forty-five-inch-long pipe sections, including both LSL sections obtained from Rochester, N.Y. and sections of commercially available soft (annealed) Type L copper tubing were either coated with epoxy or lined with PET.

Samples collected from the fill-and-dump experiments were analyzed for lead and copper to determine the effectiveness of the PET liner and epoxy to block the release of metals from the lead and copper inner pipe wall. Samples were also analyzed for TOC and for residual chlorine in samples where the extraction water initially contained chlorine. Parallel tests were performed with copper pipe specimens to determine the ability of lining and coating to produce similar results with another piping material.

Elemental analyses of a commercially available PET liner and a commercially available epoxy were performed to determine inorganic constituents that may leach into drinking water. The inorganic constituents of concern discovered in the lining or coating were monitored in samples collected from the fill-and-dump experiments.

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CHAPTER 3: METHODOLOGY

To measure the effect of the epoxy coating and PET liner on water quality, fill-and-dump experiments were performed. Multiple extraction waters and detention times were selected in order to simulate a range of field conditions experienced in usage. Parallel tests were performed with lead and copper pipes to demonstrate reproducibility with regards to the effect of linings and coatings on water quality and their ability to block metal release from two different pipe materials. Fill-and-dump tests consist of pouring water of known quality into a pipe, allowing it to stagnate under conditions similar to those that may be experienced in the field, pouring the water out, and analyzing the resultant sample for any changes to water quality. Since the quality of water in prolonged contact with a coating or lining will be influenced to a greater extent by leaching than will flowing water, fill-and-dump tests were chosen to represent a worst-case scenario. To determine possible leachates, tests were performed on the coating and lining materials prior to the fill-and-dump experiments. To ensure against experimental artifacts, the end-fitting materials used to facilitate the fill-and-dump experiments were also tested for leaching and adsorption of compounds of interest, e.g. lead, copper, antimony, and TOC. This chapter describes the methods and analytical procedures used for these experiments.

REAGENTS

Chemicals

TraceMetal™ grade concentrated nitric acid (67 – 70%, Cat. No. A509-P212, Fisher Chemical™, Pittsburg, Pa.) was used for the preservation of samples collected for metals analysis. TraceMetal™ grade concentrated hydrochloric acid (HCl) (30 – 32%, Cat. No. A508-4, Fisher Chemical™, Pittsburg, Pa.) was used to generate 1.0 and 0.1 M solutions of HCl for pH adjustment. Standards for lead analysis were made from Claritas PPT® grade lead standard (1000 mg/L, Cat. No. CLPB2-2Y, SPEX Certiprep®, Metuchen, N.J.). Standards for copper analysis were made from Claritas PPT® grade copper standard (1000 mg/L, Cat. No. CLCU2-2Y, SPEX Certiprep®, Metuchen, N.J.). Standards for antimony analysis were made from Claritas PPT® grade antimony standard (1000 mg/L, Cat. No. CLSB7-2Y, SPEX Certiprep®, Metuchen, N.J.). Laboratory-grade sodium hypochlorite solution (5.65 – 6.00%, Cat. No. SS290-1, Fisher Scientific™, Pittsburg, Pa.) was used to create chlorinated and chloraminated solutions. Except where otherwise noted, American Chemical Society (ACS) reagent-grade chemicals were used to make all other solutions, stocks, and standards.

Reagent Water

Ultra-pure, filtered and deionized 18.2 megaohm (MΩ) water was used as reagent water. To obtain reagent water, Lawrence, Kan. tap water was processed first by a reverse osmosis system (Model Elix 10, EMD Millipore, Billerica, Mass.) and then by a polishing system (Model Milli-Q® Advantage A10®, EMD Millipore, Billerica, Mass.) utilizing UV disinfection and microfiltration. This reagent water was used in all solutions, stocks, standards, and extraction waters, except where noted otherwise.

Extraction Waters

Four types of extraction waters are outlined in National Science Foundation (NSF)/American National Standards Institute (ANSI) 61 Annex B.9 (NSF International 2010) for testing drinking water components. The pH values specified for the extraction waters are 5, 6.5, 8, and 10. The waters with a pH of 5, 6.5, and 10 contain 2 mg/L available chlorine. The waters with a pH of 5, 6.5, and 8 contain a hardness of 100 mg/L as $CaCO_3$. Three extraction waters were developed for use in the fill-and-dump experiments with lined and coated pipe specimens, two of which were based on the pH 6.5 and pH 8.0 NSF/ANSI 61 waters. Instead of the pH 5 NSF/ANSI extraction water designed for metal leaching analysis, the low pH extraction water used in this study was based on the pH 6.5 NSF/ANSI extraction water since it better represents the lower pH range of drinking water in use in the United States. Dechlorinated tap water was prepared using Lawrence, Kan. tap water to represent a typical finished drinking water.

Chlorinated pH 8 Extraction Water and Chloraminated pH 8 Extraction Water

Chlorinated pH 8 extraction water was prepared based on the pH 8 NSF/ANSI extraction water for organic analysis. Reagent water was allowed to acclimate to room temperature by storing it in 4 L amber glass bottles tightly sealed with caps lined with polytetrafluoroethylene (PTFE) and placed in the same temperature-controlled room used for the fill-and-dump tests. This water was dosed with 1.00 mM reagent-grade $CaCl_2$ to obtain a hardness of 100 mg/L as $CaCO_3$. For the first sets of fill-and-dump experiments, this water was dosed with 1.00 mM $NaHCO_3$, then adjusted to pH 8.0 ± 0.1 using 1.0 and 0.1 N hydrochloric acid. Since this water was not in equilibrium with the atmosphere with respect to carbon dioxide, its pH was prone to large and variable fluctuations as the water was poured into and dumped out of the pipe specimens. Therefore, for subsequent tests, this water was prepared to have an initial composition of 0.56 mM $NaHCO_3$ and 0.44 mM NaCl, such that its initial pH would be close to 8 and it would also be close to equilibrium with the atmosphere with respect to carbon dioxide. Immediately prior to use, this extraction water was dosed with sodium hypochlorite to obtain a free chlorine concentration of 2 mg/L as Cl_2. The pH of the water was then adjusted to 8.0 ± 0.1 using 0.1 N HCl. The extraction water was prepared as needed in 4 L batches stored in 4 L amber glass bottles tightly sealed with PTFE-lined caps.

For experiments requiring chloraminated extraction water, chlorinated pH 8 extraction water was dosed with ammonium chloride at a molar concentration equal to the free chlorine level (0.0282 mM) for a Cl_2:NH_3 of approximately 5:1 by weight. At this ratio, monochloramine should be the predominant chlorine species (Equation 1).

$$NH_4^+ + OCl^- \rightarrow NH_2Cl + H_20 \qquad \text{(Equation 1)}$$

pH 6.5 Extraction Water

The pH 6.5 extraction water was prepared with reagent water that was allowed to acclimate to room temperature by storing it in 4 L amber glass bottles tightly sealed with PTFE-lined caps and placed in the same temperature-controlled used for the fill-and-dump tests. This extraction water was initially prepared by dosing the reagent water with 1.00 mM reagent-grade

$CaCl_2$ to obtain a hardness of 100 mg/L as $CaCO_3$, buffered with 1.00 mM reagent-grade $NaHCO_3$, then adjusting the pH to 6.5 immediately prior to use using 1.0 and 0.1 N HCl. For reasons similar to those stated above for the pH 8 water, subsequent batches of the pH 6.5 reagent water were prepared using 0.018 mM $NaHCO_3$ and 0.982 mM NaCl. Immediately prior to use, the pH was checked and adjusted, if necessary, using 0.1 N HCl. No chlorine was added to the pH 6.5 extraction water. The extraction water was prepared as needed in 4 L batches stored in 4 L amber glass bottles tightly sealed with PTFE-lined caps. The pH 6.5 extraction water represents a reasonable worst-case scenario for the leaching of metals due to its low pH and negligible ability to precipitate or coprecipitate metals.

Dechlorinated Tap Water

Monitored concentrations of Lawrence tap water constituents in 2011 are shown in Appendix B – City of Lawrence, Kan. Drinking Water Consumer Confidence Report. After running the tap for 5 min., a 28 L HDPE cylindrical tank (Nalgene™, Thermo-Scientific™, Waltham, Mass.) was filled with 13 – 20 L of tap water. The tap water, which is chloraminated, was dechlorinated by drop-wise addition of a 0.14 M solution of $NaHSO_3$ while stirring with a 2 ft. section of 3/4 in. HDPE tubing (Advanced Technology Products, Milford Center, Ohio, meets NSF 51 and 61 standards) until the total chlorine concentration was non-detectable (below 0.1 mg/L as Cl_2). The pH of the dechlorinated tap water (initially 8.3 – 8.4) was then adjusted to pH 8.0 ± 0.1 using 0.1 N hydrochloric acid. Before dechlorination and pH adjustment, the alkalinity of the tap water was determined. After pH adjustment, the tap water was analyzed for dissolved oxygen (DO) and samples were collected for analysis of TOC and metals.

ANALYTICAL METHODS

Strict quality assurance (QA) guidelines were developed and followed for this project. Quality control (QC) processes were incorporated into the standard operating procedure (SOP) for each analysis, and each data set was carefully reviewed to be sure QC guidelines were met and to attempt to detect any experimental or instrumental problems. For most analyses, enough sample volume was collected from the experiments to re-run any samples that did not initially meet the QC guidelines. However, in some cases, the volume of sample collected for TOC analysis was only enough to analyze one sample. If QC guidelines were not met, and no more sample was available, the data for that sample were carefully reviewed, the results were marked with a footnote indicating the deviation from the QC guidelines, and justification was given for including or excluding the results from statistical analyses.

Sample Storage and Preservation

Samples for later analysis of lead, copper, and antimony were stored at 4º C in polypropylene centrifuge tubes (15 mL, Cat. No. 05-539-12, Fisherbrand™, Pittsburg, Pa.) and preserved by adding 4 drops of nitric acid per 10 mL of sample. Samples for later analysis of hardness, sodium, and potassium were stored at 4º C in polypropylene centrifuge tubes (50 mL, Cat. No. 05-539-13, Fisherbrand™, Pittsburg, Pa.) and preserved by adding 4 drops of nitric acid per 10 mL of sample. Samples for later analysis of TOC were stored at 4º C in amber borosilicate glass vials with screw caps and PTFE-lined septa (40 mL, Cat. No. 2122-40mlT,

QEC, Beaver, W. Va.) and were at first preserved for later analysis by adding 5 drops phosphoric acid per 10 mL of sample. Storage of samples from the epoxy-coated pipe specimens for TOC analysis resulted in the precipitation of minute amounts of a white fluffy compound that likely contained phosphorous. To reduce the potential for precipitation, acid addition to preserve TOC samples collected from the PET-lined pipe sections was reduced to 1 drop per 10 mL of sample, which was still more than enough to drop the pH of the samples to below 2. Samples collected for analysis of pH, alkalinity, conductivity, chlorine, and dissolved oxygen were analyzed immediately and not stored.

Analysis of Major Water Quality Constituents

Standard Methods (APHA, AWWA, and WEF 2005) were followed for all analyses except the analysis of chlorine, which used Hach Method 8167 (Hach 2014). Alkalinity was analyzed according to Standard Method 2320 using an automatic titrator (Model DL15, Mettler Toledo, Columbus, Ohio). Dissolved oxygen (DO) was measured following Standard Method 4500-O using a DO meter (Model 5000, YSI Incorporated, Yellow Springs, Ohio). Analysis of pH was performed following Standard Method 4500-H^+ using a pH meter (Accumet™ AB15, Fisher Scientific™, Pittsburg, Pa.) equipped with a double junction pH electrode (Ag/AgCl, Orion™ 9107BN Triode™, Thermo Scientific™, Waltham, Mass.) and an automatic temperature compensation probe.

Analysis of TOC was performed using a portable TOC analyzer (Sievers Model 900, GE Instruments, Boulder, Colo.) located at the Clinton Lake Lawrence Water Treatment Plant in Lawrence, Kan. Several steps were taken for quality control. Among the collected laboratory samples, the following were also tested every time the TOC analyzer was used: a set of at least 3 calibration standards, 1 reagent blank and 1 calibration check for every 10 samples, 1 duplicate sample, 1 matrix spike, and 1 sample spiked with inorganic carbon. Five replicates were tested for each sample, with the first two omitted to minimize instrument 'memory'. The remaining replicates were required to have a relative percent standard deviation (%RSD) of ≤ 2% for TOC levels > 2000 µg/L, ≤ 3% for TOC levels ≤ 2000 µg/L, ≤ 5% for TOC levels ≤ 1000 µg/L, and ≤ 10% for TOC levels ≤ 500 µg/L. If the %RSD criteria for a data point were not met, the sample was re-analyzed until the %RSD criteria were met. If a sample was analyzed more than once for TOC and none of the results met QA guidelines, the result based on replicates having the lowest relative standard deviation (RSD) was used. The method detection limit (MDL) for TOC, based on analysis of a 1 mg/L standard on 8 different days, was 0.1 mg/L. However, caution should be exercised when drawing conclusions from low-level TOC measurements (≤ 0.30 mg/L). A small and variable amount of TOC is present in the reagent water and introduced during sample handling, and there is no universally agreed upon method of handling "TOC" associated with the instrumental response produced when ultrapure water is injected into a TOC analyzer.

Hardness was determined by analyzing samples for calcium and magnesium, assuming they were the major constituents contributing to hardness. Calcium and magnesium, along with major cations (sodium and potassium), were analyzed using Standard Method 3111 and a flame atomic absorbance spectrometer (Varian 240, Agilent Technologies, Santa Clara, Calif.). Hach Method 8167 was followed to analyze chlorine concentrations using an UV-Visible spectrometer

(DR 5000, Hach, Loveland , Colo.) and AccuVac® Ampuls for total chlorine (Hach, Loveland, Colo.).

Analysis of Metals

Standard Method 3113 (APHA, AWWA, and WEF 2005) was followed to analyze lead, copper, and chromium using an atomic absorption spectrophotometer equipped with a graphite furnace (Varian Model 120, Agilent Technologies, Santa Clara, Calif.). Ammonium di-hydrogen phosphate (5,000 mg/L) and palladium (500 mg/L) were used as matrix modifiers in the analysis of lead and copper, respectively. Several QC samples were analyzed with every 20 samples in each run of the instrument, including: duplicate sample, laboratory-fortified blank (LFB), laboratory-fortified matrix (LFM), and reagent blank. To meet QC guidelines, the concentrations of the LFM and LFB had to be measured within 20% of their actual value and the reagent blank had to be below the MDL for the metal measured. Furthermore, the %RSD had to be less than 5% for lead results > 2 ppb and less than 10% for lead results < 2 ppb. The %RSD had to be less than 5% for copper results > 1 ppb and less than 10% for copper results < 1 ppb. If any of these QC guidelines were not met, corrective measures were taken, including: instrument recalibration, re-analysis of a failed sample, or re-analysis of the entire batch of samples. The MDLs for lead and copper using these methods were 0.5 μg/L and 0.25 μg/L, respectively.

Antimony analysis was performed using inductively coupled plasma mass spectrometry (ICP-MS) (PlasmaQuad II+XS, VG Elemental, Thermo Scientific™, Waltham, Mass.). At least 5 standards were analyzed at the beginning and end of each run of the instrument to establish and check the calibration curve. A continuing calibration check and LFM were analyzed with every 10 samples. To meet QC guidelines, the LFM and continuing calibration check had to be measured within 20% of their actual value. The MDL for antimony using these methods was 0.06 μg/L.

PREPARATION OF PIPE SPECIMENS

Pipe Preparation

Approximately 100 year old lead pipe sections, 3.5 to 4 ft. long with 5/8 in. inner diameter (ID), with the exception of two having a ½ in. ID, were procured from a water utility (the Rochester, N.Y. Water Bureau) that had recently removed them from service. Some pipes were excavated, but most were pulled from the ground by inserting a chain through the pipe's length and securing it on the end. This process has the potential to damage the inner pipe as the chain rubs against it. The whole pipe can also be damaged due to the compressive and tensile stresses that pulling the pipe through a resistant soil can place on the ductile lead. Consequently, the pipes were inspected for damage once received, and those showing signs of damage were set aside and not used. The external surfaces of the pipes were then cleaned using tap water to remove excess dirt. After drying, the pipes were wrapped with duct tape to minimize potential contamination from handling the outer surfaces. To prevent extraction water from coming into contact with the end of the lead pipe while being poured out over the lip of the pipe, stainless steel (SS) end fittings were installed on both ends of each pipe, as shown in Figure 1.

The ends of each LSL pipe specimen were reamed and then threaded with a ½ in. nominal die. After wrapping three times with PTFE tape (Poly-Temp® XHD, Anti-Seize Technology, Inc., Franklin Park, Ill., meeting C.I.D. Spec A-A-58092), ½ in. ID (nom., about 5/8 in. actual ID) by 2-½ in.-long 316 SS threaded pipe nipples (Part No. 1XAB2, Grainger, www.grainger.com, meeting ASTM Standard A733) were installed on both ends of each LSL pipe specimen. Before installation, the nipples were scrubbed with a stiff brush to remove any remaining grease residue from the manufacturing process, rinsed with warm tap water, then agitated and soaked in hexane for at least 5 minutes. SS hose clamps (Breeze Aero-Seal® Model 200 12H, Norma Group, Auburn Hills, Mich.) were installed over the pipe ends and nipples to tightly secure the nipples and decrease leakage. The pipes were then wrapped in foam insulation to minimize any damage from shipping and handling and to provide an extra layer of protection from surface lead contamination.

Figure 1: Image of fully prepared pipe specimens. On the top, a LSL with threaded SS nipples and silicone stoppers. On the bottom, a CSL with unthreaded SS pipe nipples connected with polypropylene compression fittings and fitted with HDPE stoppers.

Copper pipes with a nominal size of 5/8 in. (and an actual ID slightly larger than 5/8 in.) were cut into 45 in. long sections from coils of Type L, potable water grade 'soft' annealed copper tubing (ASTM B-88, Great Lakes Copper Inc., London, Ontario, Canada) and carefully straightened to avoid kinking. The ends were reamed and sanded to remove burrs and to improve contact with end-fittings. Polypropylene compression fittings (Tube Bulkhead Union, JACO Manufacturing Company, Berea, Ohio) were soaked and rinsed in reagent water and installed on both ends of the pipe. Unthreaded nipples were made from SS pipe (½ in. nom. ID, about 5/8 in actual ID; Part No. 4NTN6, ordered from Grainger, www.grainger.com, meeting ASTM A269/A213 and ASME SA213 standards) that was cut into 3 in. sections. The unthreaded SS nipples were then scrubbed with a stiff brush then rinsed with tap water and finally hexane (to remove any residual cutting oil) before being inserted into the polypropylene fittings. The pipes were then wrapped with foam insulation to minimize damage from shipping and handling and to cover all exposed copper.

Epoxy Coating Application

There were 18 pipe sections selected for epoxy coating: 8 lead pipe sections with an actual ID of approximately 5/8 in., 2 lead pipe sections with an actual ID of approximately ½ in., and 8 copper pipe sections with a nominal size of 5/8 in. These pipe sections were shipped to a Nu Flow Technologies facility located in San Diego, Calif. where the epoxy application was performed as it would be in the field. The same equipment used in the field was used for this

application, including: large air compressor, air filtration system, heating elements, hoses, connectors, and sand hopper.

The pipes were set up horizontally on a wall in 9 pairs, with 2 pipes of identical material and similar ID in each pair. This configuration allowed the application process to go more quickly than if each pipe were coated individually, as preparing and moving the equipment is much more time intensive than the actual coating process. To connect the lead pipe specimens, brass fittings were used. The SS nipples were removed from the copper pipes and replaced with transparent acrylic pipe in order to connect them to each other and with the connectors used by Nu Flow Technologies to hook up to their equipment.

A large air compressor was situated outside the building, approximately 50 yards away. A hose went from the compressor to a company truck that housed an air filtration system. This system was designed to remove any oil and water introduced by the compressor. From the filter, a hose went inside to be connected to either the sand hopper or a heating element. From the sand hopper, heating element, or water faucet, a 1-½ in. hose ran to the copper or lead pipe sections to be coated. Exiting the pipe section, another 1-½ in. hose was used to complete the system and allow for waste collection.

The first step in the coating process was to sandblast the inside of the pipes. The goal was to give the epoxy a fresh, rough surface to adhere to. If done properly, the surface should look similar to sand paper, commonly referred to as an 'anchor tooth' pattern. Approximately one cup of sand was used for each blast (of about 8 total) applied to the copper pipes. The sand was added manually for the copper pipes to reduce the pressure load on the glued connectors that Nu Flow Technologies used on their own equipment to connect to the copper pipes. The lead pipes were able to withstand the pressure required to use the hopper, and were given about ten 3 or 4-second-long shots of sand. When the desired anchor tooth pattern was thought to have been attained, a small camera mounted on a long USB cable and connected to a computer was used to inspect the inner wall texture. If the texture did not look quite right, further sandblasting was performed until the proper texture was achieved.

The pipes were then flushed with tap water to remove lead dust. In the field, copper pipes are flushed only if there is lead solder present. For this study, all pipes were flushed, including the copper pipes. A second flush was performed in the opposite direction to rinse threads, fittings, and transitions evenly. In the field, the flush water is typically drawn from a hydrant or pump truck and a higher pressure is used. After flushing, the pipes were dried using heated, filtered air. The air was heated using a heating element located between the filter and pipe. An infrared thermometer was used to measure the temperature of the outer pipe. Feeling the heat by touch was also employed to get a good sense of when the pipe had finished drying.

The two-part epoxy was created by mixing 70% part A and 30% part B, by weight. The amount of epoxy mixed was the amount the workers thought they could install before the epoxy's working time expired. The first batch proved sufficient to coat all 8 copper pipes. Immediately prior to application of epoxy, the pipes were heated once again to improve the epoxy's adherence and prolong the epoxy's working time. The pipes were coated in pairs. Enough epoxy to coat two pipes was poured into a 4 ft. shot tube, which was coiled into a single

loop that allowed the epoxy to pool at the bottom as it was connected to the pipe. The other end of the epoxy filled loop was connected to the air flow system. Air pressure and flow were applied to spread the epoxy along the inner walls of the pipe. The amount of air pressure and flow were determined by the inner diameter and length of the pipe. Looking through the acrylic pipe makes the coating process easy to see, as shown in Figure 2. The epoxy is not a slug of liquid being pushed through the pipe. Instead, a channel of air flows through the liquid, creating a vortex. The epoxy is then spun into the wall of the pipe as a uniform leading edge moves along the length of the pipe.

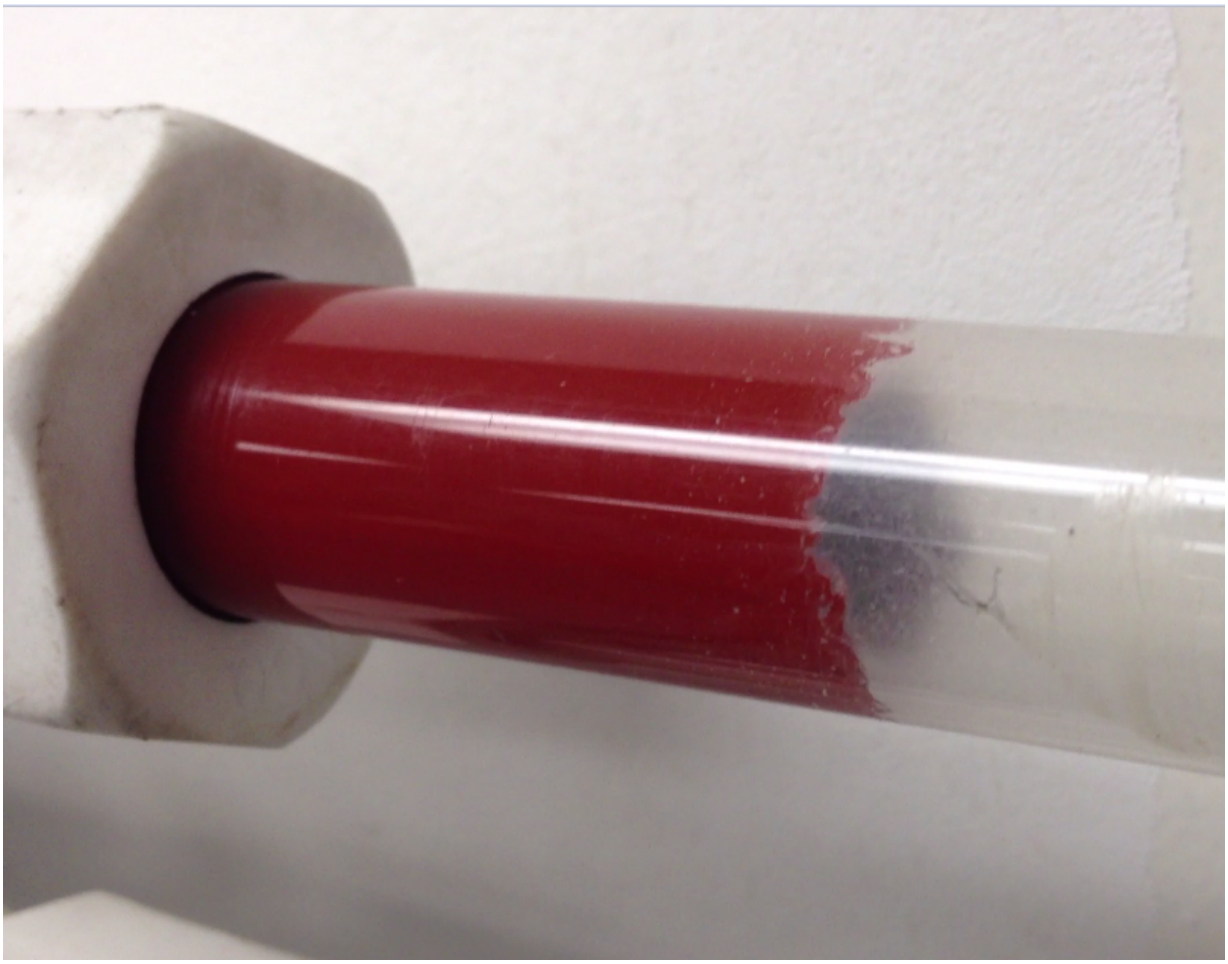

Figure 2: Image of epoxy coating as it is being installed through transparent acrylic pipe

Once the epoxy was installed, all connections from the Nu Flow equipment were unhooked, which must be done before the epoxy dries. The unthreaded SS nipples were re-attached to the copper pipes. In the field, air would be blown through the pipes for an additional 2 – 3 hours, expediting curing and minimizing pooling effects. As this demonstration involved 9 separate connections (one for each pair of pipes), the air flow could not be reasonably continued for that long. Therefore, the pipes were hung vertically on a rack and allowed to cure. One coated and one uncoated LSL (used as a control) are depicted in Figure 3 to compare the pipes before and after epoxy coating.

Figure 3: Image comparing an epoxy-coated LSL (left) to an uncoated LSL (right)

PET Liner Installation

The PET liner installation was not witnessed in person, but consists of inserting an unexpanded liner into the pipe then applying pressure in the form of hot water or air to expand the liner until it fits tightly against the inner wall of the pipe, as explained in further detail in Chapter 2. A sample of unexpanded PET liner, acquired from Flow-Liner Systems, can be seen in Figure 4 protruding from an unthreaded SS pipe nipple attached to a CSL specimen. One lined and one unlined LSL (used as a control) are depicted in Figure 5 to compare the pipes before and after the PET liner installation.

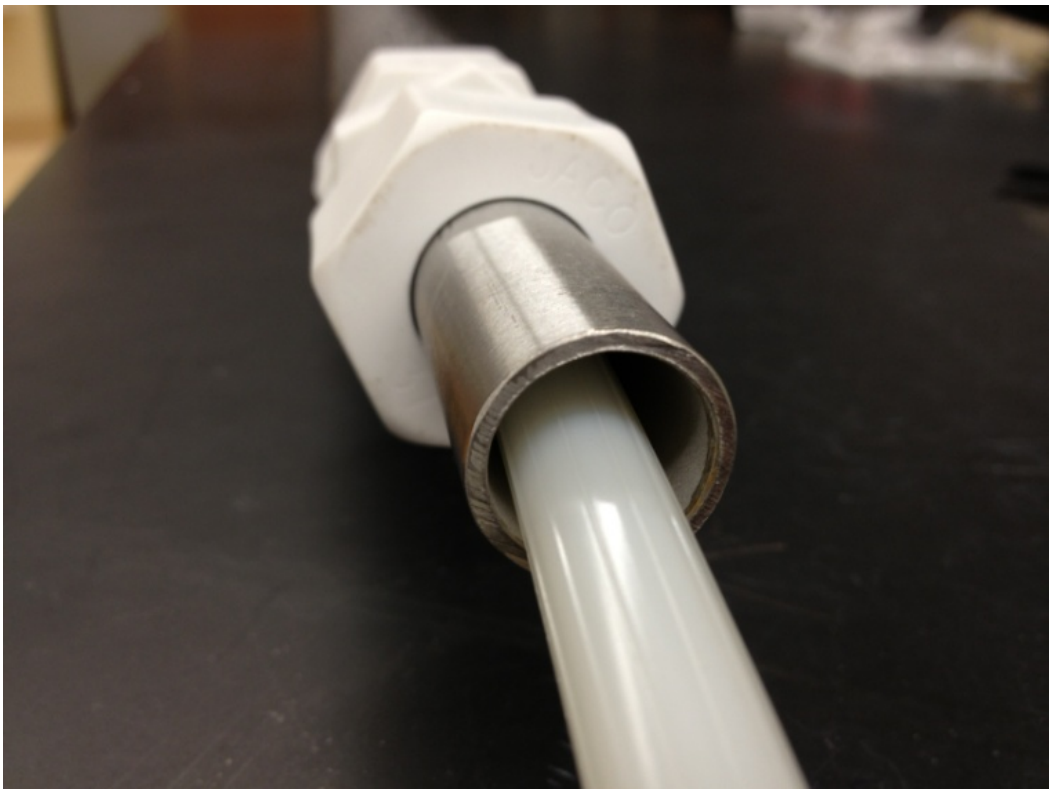

Figure 4: Image of an unexpanded PET liner being inserted into an unthreaded SS nipple attached to a CSL specimen

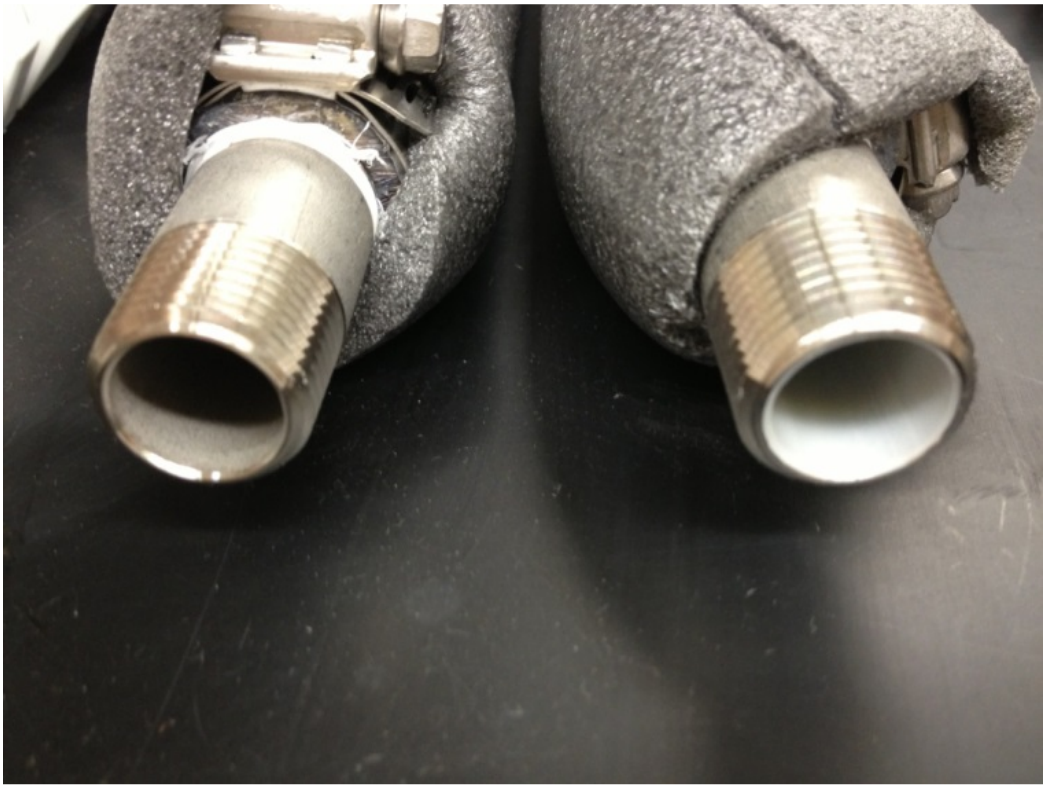

Figure 5: Image comparing an unlined LSL (left) to a PET-lined LSL (right)

EXPERIMENTAL PROCEDURES

Elemental Analysis of Epoxy

A two part, red-colored epoxy was acquired from Nu Flow Technologies for a series of bench-scale tests. The epoxy is NSF 61 certified and potable water grade. To create the epoxy, Part A and Part B were combined in a 70% and 30% mixture by weight, respectively. The epoxy can then be applied as a coating in a working time of several minutes. With the application of heat, as is performed in typical applications, the working time can be extended. According to the epoxy's Material Safety and Data Sheets (MSDS), Part A contains 5% iron oxide by weight, which is assumed to be the source of the red color. Part B contains <10% triethylenetetramine (TETA), a primary aliphatic polyamine used as a hardener and curing agent (Nu Flow Technologies 2011a; 2011b).

Further elemental analysis is beneficial to determine the presence of any unknown compounds that may leach into drinking water. The epoxy was digested to facilitate analysis for lead, copper, and chromium, elements likely to be present in both the pipe specimens and in the end-fittings used in the fill-and-dump tests, which could potentially compromise the experimental results or complicate their interpretation. Approximately 3 grams of epoxy were mixed and allowed to cure as a thin layer on a polystyrene weighing dish (Hexagonal Type, Fisherbrand™, Pittsburg, Pa.). The coating was then carefully chipped away from the plate and placed in an Erlenmeyer flask with 9 mL nitric acid and 3 mL of hydrochloric acid. The total mass of the cured epoxy chips was 1.1 grams. The flask was then topped with a lens and placed on a hot plate set to a temperature of 85^{o}C. The mixture was allowed to digest for 24 h after which another 10 mL nitric acid, 4 mL hydrochloric acid, and 5 mL of 30% hydrogen peroxide were added. The temperature was then raised to 90^{o}C. Peroxide was added periodically to quicken the digestion for a total of 134 mL. Nitric and hydrochloric acids were also added periodically for totals of 27 mL and 10 mL, respectively. The epoxy was never fully digested, although a significant portion of the mass bleached from red to yellow. The leachate was tested for lead, copper, and chromium using graphite furnace atomic adsorption spectrometry (GFAAS). A scan for other elements of concern was also performed using ICP-MS.

Elemental Analysis of PET Liner

A multi-elemental analysis of the PET liner was conducted using ICP-MS. The PET liner was acquired from Flow-Liner Systems (Zanesville, Ohio) and is NSF 61 and 61G certified. Initially, an ablation technique was used to draw the samples into the ICP-MS instrument directly from the solid liner by changing it into an aerosol with a laser. Once the aerosol is ionized by argon plasma and enters the spectrometer, it is separated by a quadrupole that allows ions of only a single mass-to-charge ratio to hit the detector. The detector then counts the ions it detects per second. With the particular instrument used in this test, the detector counts continuously as the sample is being processed. If the sample has the specific element being tested, a noticeable jump in ion counts can be viewed as the ions reach the detector, approximately one minute after the sample is drawn. Since the amount of liner drawn from the solid by the laser is unknown, the results using this method were not quantitative, and were used only to determine which elements were present in the liner material. Subsequently, a digestion

was performed on a known mass of liner and the digestate was diluted in a known volume of solution to provide a sample from which ICP-MS analysis could produce quantitative results.

PET liner was digested in triplicate using a microwave digester (Multiwave 3000, Anton Paar, Ashland, Va.). Approximately 0.1 – 0.3 mg of the PET was cut into approximately 2 mm by 2 mm pieces and placed into a solution of 10 mL 65% nitric acid and 2 mL 30-32% hydrochloric acid. A control solution containing only the combined nitric and hydrochloric acid mixture was also prepared. The solutions were placed in acid washed PTFE vessels and into a microwave digester. Two stages were used in the digestion. The first stage introduced elevated temperature and pressure, achieved by setting the microwave power output at 400 W, with a pressure increase rate of 0.3 bar/s for 15 min. This condition was held for 60 min. The second 'cool down' stage was set to a power output of 0 W but maintained pressure for 20 min. After digestion, the vessels were rinsed into a volumetric flask and diluted to 100 mL with reagent water. Solids were present in the samples, which were filtered with a 0.45 μm PTFE syringe filter (Fisherbrand™, Pittsburg, Pa.) before ICP-MS analysis. The results from the ICP-MS analysis quantified the concentration of tested elements in each sample. These results were divided by their respective known PET liner concentrations present in each sample and averaged to determine the mean concentration in the PET. The calculated concentrations may be a low estimate if the solids removed through filtration contained the elements tested. However, the calculated concentrations could be reasonably close to the true values if the application of heat, pressure, and acid was enough to leach most of the elements tested from the plastic.

Physical Characteristics of Epoxy Coating, PET Liner, and Pipe Specimens

The volume of each LSL was determined before and after coating or lining. Each LSL was stoppered at one end and filled with reagent water. The reagent water was then poured into a tared beaker to determine its mass, which was used to calculate the volume. Since the copper used to make each CSL was uniform, from the same coil, and cut to the same length, the (nearly identical) calculated volumes of three CSLs were averaged to determine the initial volume of each of the CSL specimens. Since the CSLs used in the PET-lined experiments were created from a different coil than used in the epoxy-coated experiments, the average CSL volume for the PET-lined CSL specimens was calculated from three new CSLs. The volume of each PET-lined or epoxy-coated CSL was individually determined by filling it with water after stoppering one end, then dumping out and weighing the water.

The length of each pipe specimen was measured and divided into the calculated volume to determine the average inner cross-sectional surface area and average inner diameter of each pipe specimen. The calculated average inner diameter in each coated or lined pipe specimen was subtracted from its pre-coated or pre-lined average inner diameter, then divided by two to determine the average coating or lining thickness. The average inner diameter was used to calculate the inner circumference then multiplied by the pipe's length to determine the inner surface area of each pipe.

Fill-and-Dump Experiments

After the pipes were coated or lined, they were shipped back to the laboratory in Lawrence, Kan. where the fill-and-dump experiments were performed. Approximately 72 hours passed between the epoxy application and the initial fill-and-dump experiments began, allowing the epoxy to cure.

In the field, coating and lining installers typically flush the pipes after installation and before putting the pipes back into use. To simulate this practice, the pipes were flushed in the laboratory after they were received from the manufacturers. Groups of two to four pipes were connected in series and flushed with tap water for 15 min. When an unlined and uncoated control section was included in the pipe manifold, it was placed last according to the direction of flow to avoid contaminating a lined or coated pipe with metals released from a control pipe. After flushing, and immediately before filling a pipe with extraction water, it was first rinsed with approximately 100 mL of the desired extraction water to rinse away any tap water remaining from flushing.

The initial fill-and-dump tests on each lining or coating material were designed to determine the effects of freshly cured epoxy or freshly installed liners on water quality over periods of time that might reasonably be encountered in actual service. Detention times used in the fill-and-dump experiments were 6 hours, 24 hours, 4 days, 7 days, and 10 days. The 6 hour detention time was based on current LCR monitoring criteria that require a stagnation period of at least 6 hours to represent the time water is typically left standing in a service line overnight before water use continues again in the morning. Longer periods of time (24 hours to 10 days) were used for two purposes: 1) to examine the potential release of specific constituents over time; and 2) to represent reasonable worst-case scenarios, such as members of a household on a week-long trip.

When the desired detention time was reached for a given pipe section, the extraction water was immediately and carefully poured into a glass beaker that had previously been acid washed, rinsed with reagent water, and dried. From this glass beaker, samples were immediately divided for analysis of pH, lead, copper, and TOC, and for analysis of residual chlorine in cases where the extraction water contained chlorine. Samples collected from PET-lined pipes were also analyzed for antimony. Analysis of pH and chlorine residual was performed immediately, while samples for metals and TOC were preserved and stored for later analysis as described above.

Samples of the extraction water were also collected immediately before the fill-and-dump experiments to determine initial concentrations of any constituents analyzed for in samples collected from pipe specimens. The extraction water was also analyzed for chlorine residual immediately after fill-and-dump tests, in cases where the extraction water contained chlorine. Chlorinated extraction waters were stored in tightly capped 4 L amber-glass bottles next to the pipe specimens to keep them at the same temperature. Amber glass was used to reduce exposure to light, which could potentially affect the free chlorine residual.

Procedure for Fill-and-Dump Experiments Using Epoxy-Coated Pipes

Selected epoxy-coated pipes and control pipes (one of each type) were filled with dechlorinated tap water and held for 6 hours, 24 hours, or 4 days. Two pipes of each type (lead and copper) were held for 6 hours as duplicates. Once the pipes that held dechlorinated tap water for 24 hours were emptied, they were immediately filled again with dechlorinated tap water and held for 10 days. A second set of coated pipes were filled with chlorinated pH 8 extraction water employing the same detention times as for those filled with dechlorinated tap water, but without a duplicate.

Pipes previously exposed with either dechlorinated tap water or chlorinated pH 8 extraction water were selected, filled with pH 6.5 extraction water, and held for 6 hours. The pipes selected include: the control pipes (one of each type), the duplicate pipes that held dechlorinated tap water, and pipes (one of each type) that held chlorinated pH 8 extraction water. Once emptied, the pipes were then filled with pH 6.5 extraction water again and held for 7 days. A full testing matrix can be found in Table 19 of Appendix D. After this first set of experiments all pipes except for one coated lead and one coated copper pipe were allowed to drip dry and were then stored air tight with polypropylene caps. The two pipes not stored dry were filled with reagent water and capped with silicone stoppers (LabPure® PX 18D and 21D, Saint-Gobain Performance Plastics, Portage, Wis.). Every 7 days, the wet-stored pipes were emptied and filled with fresh reagent water.

Approximately 7 months after the first set of experiments, additional fill-and-dump tests were performed using chlorinated pH 8 extraction water and detention times of 6 hours, 24 hours, and 7 days. Among the pipes selected were the 2 wet-stored pipes, 2 dry-stored coated pipes, 2 dry-stored coated pipes never used in the first set of experiments, and the uncoated lead and copper control pipes. Before being used in this second set of experiments, the two coated pipes never used in the first set of experiments were flushed (for the first time) for 15 minutes using tap water. The other, previously used pipes were not flushed again prior to the second set of experiments. After this second set of experiments, all tested pipes underwent the 15-minute flushing procedure once more and were subjected to a third round of testing.

A set of epoxy-coated lead and copper pipes, previously filled with chlorinated pH 8 extraction water and held for 6 hours and then 10 days, were selected for a series of sequential one-hour fill-and-dump tests using chlorinated pH 8 extraction water to determine the effect on chlorine demand after repeated exposure to chlorinated water. After 9 one-hour tests, the pipes were filled with chlorinated pH 8 extraction water dosed with 100 mg/L free chlorine as Cl_2 and dumped after three hours in a test modeled after the slug method for disinfecting water mains (AWWA 2005). The samples were then analyzed to determine the remaining concentration of free chlorine. This test was repeated three times in an effort to exhaust the epoxy coating's chlorine demand. Another series of 6 1-hour fill-and-dump experiments with chlorinated pH 8 extraction water dosed with 2 mg/L free chlorine as Cl_2 were performed again on the 'disinfected' pipes and the samples were immediately analyzed to determine the remaining concentration of free chlorine.

To test for combined chlorine demand, epoxy-coated pipe specimens were filled with chloraminated pH 8 extraction water and held for 6 hours, 24 hours, and 4 days. The extraction water samples were analyzed immediately after dumping to determine the remaining concentration of combined chlorine.

Procedure for Fill-and-Dump Experiments Using PET-Lined Pipes

Selected PET-lined pipes and control pipes (one of each type) were filled with dechlorinated tap water and held for 6 hours, 24 hours, and 4 days. Two more pipes (one of each type) were filled with dechlorinated tap water and held to duplicate the 6 hour detention time. A second set of lined pipes were filled with chlorinated pH 8 extraction water employing the same detention times as for those filled with dechlorinated tap water, but without a duplicate.

Pipes previously exposed to either dechlorinated tap water or chlorinated pH 8 extraction water were selected, filled with pH 6.5 extraction water, and held for 6 hours. The pipes selected include: the control pipes (one of each type), the duplicate pipes that held dechlorinated tap water, and pipes (one of each type) that held chlorinated pH 8 extraction water. Once emptied, the pipes were then filled with pH 6.5 extraction water again and held for 4 days. A full testing matrix can be found in Table 28 of Appendix D. After this first set of experiments, all pipes were allowed to drip dry and stored air tight with polypropylene caps.

During the flushing procedure, it was noted that water was leaking from the pipe end-fittings. For lined lead pipes that leaked, water was seen dripping from where the SS nipple threaded into the lead pipe. For lined copper pipes that leaked, water was seen dripping from where the unthreaded SS nipple was inserted into the polypropylene compression fitting. Since the inner wall of these connections was sealed with PET liner, the leaks indicated that water was traveling between the pipe wall and the liner itself. Despite this leakage, the first set of experiments outlined in the paragraph above was carried out similarly to the experiments performed with epoxy-coated pipes. For the second set of experiments involving the PET-lined pipes, however, corrective action was taken to reduce the potential for lead and copper contamination posed by the water traveling between the liner and pipe wall.

In this second set of experiments, three sets of lined lead and copper pipes exposed to dechlorinated tap and pH 6.5 extraction waters, one set exposed to pH 6.5 and pH 8 chlorinated extraction waters, and one set never exposed to any waters were selected for re-testing. First, the SS nipples were carefully removed, leaving behind exposed PET liner extending approximately 2.5 – 3 in. from each end of each lead or copper pipe. The ends of newly exposed PET liner on many of the pipes were found to be wet and/or to have heavy deposits of a dark brown substance that had accumulated between the liner and SS nipple. The outer surface of each exposed liner end was carefully cleaned with a new laboratory wipe (Wypall Type L30, Kimberly-Clark, Roswell, Ga.) starting on the outer rim before wiping the rest of the outer surface. A fresh clean laboratory wipe moistened with 0.5% HCl was then applied in the same manner. Another fresh clean laboratory wipe moistened with 0.5% HCl was then applied to the inner liner wall, extending approximately 1 in. into the liner. The entire wiping procedure was performed once more with clean laboratory cloths and reagent water. The pipes were then flushed with two 150 mL volumes of pH 6.5 extraction water, once from each end. Along with the unlined control

pipes, these altered pipes were exposed to pH 6.5 extraction water for 6 hours, then 4 days. One set of the altered PET-lined lead and copper pipes and an unexpanded section of PET liner were selected for a series of sequential 24-hour fill-and-dump tests using chlorinated pH 8 extraction water to determine the effect on chlorine demand.

Experimental Artifacts of End-Fitting Materials

A series of tests were performed to assure the materials used in the preparation of pipe specimens would not interfere with water sample analysis. In coated and lined pipe sections, the extraction water would be in direct contact with the ends of silicone stoppers or HDPE stoppers (Type 16, Kimble Chase, Vineland, NJ), respectively. Extraction water in uncoated and unlined pipe sections (controls) would also be in contact with the SS pipe nipples and potentially with small amounts of PTFE thread tape in the case of LSL pipe sections. Furthermore, extraction water from coated and lined pipe sections would have brief contact with the SS pipe nipples as the water was poured out of the pipe into a beaker. Consequently, SS pipe nipples, silicone and HDPE stoppers, and PTFE tape were all tested to determine their ability to leach or adsorb lead, copper, and antimony. Each type of stopper (silicone and HDPE) was also tested for chlorine demand and for leaching of TOC.

Type 316 SS pipe nipples (threaded and unthreaded) were procured and tested for adsorption and leaching by filling them with 0.072 μM solutions of both lead and copper. Two nipples of each type (threaded and unthreaded) were tested along with control samples that did not come into contact with SS. Additionally, one nipple of each type was filled with pure reagent water. After 7 days, the water was analyzed using GFAAS. Lead concentrations remained within 2% of the control levels in both types of nipples. Reagent water exposed to both types of nipples contained no detectable amount of lead ($\leq$ 0.5 μg/L). Copper levels in the 0.072 μM lead and copper solution (4.6 μg/L Cu) increased an average of 20 μg/L in the threaded pipe nipples and 14 μg/L in the unthreaded pipe nipples. Reagent water held in the threaded pipe nipple contained 2.3 μg/L of copper while reagent water held in the unthreaded pipe nipple contained 8.3 μg/L. These data, shown in Table 14 and Table 15 in Appendix C, suggest that small amounts of copper were leaching from the SS nipples. However, the amount of copper leaching from SS nipples was substantially less than that from copper tubing, as described in Chapter 4. Furthermore, if the liner or coating is successful at blocking metal leaching, then copper would only leach from the unlined or uncoated SS nipples in the control pipe sections.

The experiment described above was performed again with a 10 μg/L solution of antimony. The antimony concentrations in waters exposed to threaded and unthreaded SS pipe nipples decreased 26% and 10%, respectively, indicating a small amount of adsorption. The data collected from these tests can be seen in Table 16 in Appendix C. As with the leaching of copper, antimony adsorption would not be expected to significantly impact the concentration of antimony found in extraction water drawn from lined or unlined pipe specimens.

Results from a laser ablation ICP-MS test on silicone stoppers indicated no detectable concentrations of lead, copper, or antimony in the stopper itself; therefore, leaching of these elements from the silicone stoppers is not expected to be a concern. To test adsorption, a

solution with equal concentrations of copper and lead (approximately 0.072 μM) was made with tap water. Different amounts of silicone stopper material, with known surface areas, were placed in aliquots of the lead and copper solution and allowed to stand (unstirred). After 7 days, lead and copper concentrations remained within 3% and 1%, respectively, at a silicone-stopper-surface-area to water-volume ratio equal to the ratio seen by the extraction water in the fill-and-dump experiments. It was determined that the silicone stoppers were compatible with the goals of the fill-and-dump experiments to be done on epoxy-coated pipe specimens, and would not leach or adsorb significant amount of lead or copper. Because silicone was found to significantly adsorb phthalate esters (by Rachael F. Lane, who was studying leaching of phthalate esters and other organic compounds in the same experiments), a different material was needed for the fill-and-dump experiments conducted on the PET-lined pipe specimens. HDPE stopper area to water volume ratios far greater than seen in the fill-and-dump experiments were used in tests for lead, copper, and antimony leaching and adsorption. HDPE stoppers were exposed to 0.072 μM solutions of lead and copper and held for 7 days. Lead and copper concentrations remained within 5% of the original concentration.

PTFE tape was tested for its ability to adsorb or leach lead, copper, and antimony in the same manner as the tests for the HDPE stoppers. Approximately 1 cm^2 of PTFE tape was placed in a 15 mL of a 0.072 μM solution of both lead and copper and held for 7 days. Lead and copper levels remained within 6% of the original solution to which the PTFE tape was exposed. Antimony concentrations in the solution exposed to PTFE tape remained within 5% of the initial concentration (10 μg/L).

Silicone and HDPE stoppers were tested for free chlorine demand by exposing them to chlorinated pH 8 extraction water (initially 2.03 mg/L free chlorine as Cl_2) held in 600 mL acid-cleaned glass beakers. Six beakers were filled with 400 mL extraction water; two beakers contained one silicone stopper each, two beakers contained one HDPE stopper each, and the remaining two beakers were used as controls. The beakers were covered with sealing film (Parafilm®, Sigma-Aldrich Corp., St. Louis, MO) and stored in the dark. After 24 hours, the average free chlorine concentrations in extraction waters exposed to either a HDPE stopper or a silicone stopper remained within 4% of the average free chlorine concentrations in both the glass-beaker controls (1.96 mg/L as Cl_2) and the 4 L amber glass bottles (with PTFE-lined caps) used to prepare and store the extraction water (1.94 mg/L as Cl_2). Table 17 in Appendix C shows the data collected from the chlorine demand tests. Thus, the stoppers did not exert significant chlorine demand.

Silicone and HDPE stoppers were tested for TOC leaching by exposing them to chlorinated pH 8 extraction water in 600 mL acid-cleaned glass beakers. Six beakers were filled with 400 mL extraction water; two beakers contained one silicone stopper each, two beakers contained one HDPE stopper each, and the remaining two beakers were used as controls. The beakers were covered with sealing film (Parafilm®, Sigma-Aldrich Corp., St. Louis, MO) and stored in the dark. After 24 hours, the TOC concentrations in extraction water exposed to silicone and HDPE stoppers remained, on average, within 0.06 mg/L of the glass-beaker control samples, a concentration less than the MDL. The data are shown in Table 18 in Appendix C. Thus, both types of stoppers were determined to not leach a significant amount of TOC. Due to the very low initial concentrations of TOC, essentially only a background level, it was not

possible to conclude from this experiment whether or not the stoppers were capable of adsorbing a significant amount of TOC from water having a higher initial TOC concentration, such as dechlorinated tap water.

Data Handling and Statistical Analysis

The first step in examining a number of the key data sets presented in Chapter 4 (Results and Discussion) was to calculate the means and standard deviations (± SD) of various subsets of data points, with n being the number of values in a given subset. The data subsets were chosen to examine the effects of selected variables such as holding time, coated versus uncoated pipe sections, or differences among extraction waters. Grubb's test ($\alpha = 0.05$) was used to determine statistical outliers. If a data point was determined to be a statistical outlier, the data point was omitted from further statistical analysis.

Some analytes were present in one of the extraction waters. For example, dechlorinated tap water contained TOC and trace amounts of both copper and antimony. In such cases, to compare diffences among extraction waters or to group together data from different extraction waters, data analysis was based on the increase in the concentration of the analyte rather than the measured concentration. The increase in concentration in an individual sample was determined by subtracting the initial concentration in the extraction water from the concentration measured in the sample following a fill-and-dump experiment. If the initial concentration measured in the extraction water was below the method detection limit (MDL), any sample concentration measured (above the MDL) was assumed to be an increase, and no correction was made to the measured value in such cases.

T-tests were used to determine whether the means of two sets or subsets of data were significantly different. First, the variances of the two data sets were examined using an F-test, to determine if they were statistically different by rejecting the null hypothesis that the variances were equal if the p-value was less than the α-value (0.05). If the null hypothesis of the F-test was rejected, the subsequent t-test would assume unequal variance. If the null hypothesis of the F-test was not rejected, the subsequent t-test would assume equal variance. The means of the two data sets were determined to be statistically different, using a two-tailed t-test, by rejecting the null hypothesis that the means were equal if the p-value was less than the α-value (0.05). The mean of one data set was determined to be statistically greater than the mean of another data set (or greater than the concentration in the control samples) by using a one-tailed t-test and rejecting the null hypothesis that the means were equal if the p-value was less than the α-value (0.05). The significance of the relationship between two variables was evaluated by performing a regression analysis and rejecting the null hypothesis (slope = 0; not statically significant) if the p-value was less than the α-value (0.05).

Box plots are used to display the range of lead, copper, and antimony concentrations measured in samples. The boxes combined represent the middle 50% of the data points and the line separating the boxes represents the median value. The top and bottom error bars represent the top and bottom 25% of values, respectively, with the end-caps of the error bars representing the maximum and minimum values. To display values below the MDL, a value of 0.5 times the MDL was used. Variables such as extraction water composition, number of times a given pipe

specimen was exposed, holding times, etc., were often not separated out in a given plot. Box plots were used primarily to graphically display the range of concentrations measured in broad subsets of samples, and not necessarily for other statistical purposes. Therefore, caution should be exercised in interpreting them, to avoid drawing conclusions not supported by the data.

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CHAPTER 4: RESULTS AND DISCUSSION

EPOXY-COATED PIPE SPECIMENS

Elemental Analysis of Epoxy

Copper concentrations in the digested epoxy were approximately 8 mg Cu/kg epoxy. Lead and chromium were not detected above a detection level of 0.1 mg Pb/kg epoxy and 0.1 mg Cr/kg epoxy, respectively, in GFAAS analysis of the digested epoxy. ICP-MS analysis of the digested epoxy confirmed the presence of copper but identified no other elements of concern.

Physical Coating Characteristics

Pipe measurements were taken, with the end fittings in place, prior to and after coating to determine physical coating and pipe characteristics. The results are summarized below in Table 1. The epoxy volume and thickness applied varied, more so for the LSLs than for the CSLs. During installation, two pipes were connected at a time to speed up the process. With the same applied air pressure being used to install the epoxy, two pipes of different diameter would experience different flow rates. Since the applied epoxy thickness is dictated, in part, by the air flow rate, it would vary between two pipes of different diameter. Initial internal diameters varied in the uncoated LSLs by as much as 2 mm in some cases. Furthermore, when the pipes were hung vertically to dry soon after the coating was applied, wet epoxy was allowed to drip out. If more epoxy dripped out of some pipes than others, this could also help explain the variance in epoxy volume and thicknesses among different LSLs. No measurements were taken from the two epoxy-coated LSLs with a ½ in. nominal ID, which were not used in the experiments described below.

Extraction Waters

Important water quality parameters of the extraction waters used in the epoxy-coated pipe experiments are summarized in Table 2. All values for dechlorinated tap water parameters, except for initial pH and alkalinity, were measured on samples taken after dechlorination and pH adjustment with 0.1 M HCl. The pH was subsequently adjusted to 8.0 and the alkalinity was lowered due to the addition of HCl. The parameters are within the ranges seen in 2011 in Lawrence, Kan. tap water except for alkalinity, potassium, hardness, and copper concentrations. However, the values measured in the dechlorinated tap water are reasonably close to those reported for 2011 considering the changes in surface water quality that can occur from one year or one season to the next.

Table 1: Physical pipe and coating data before and after epoxy coating

Type of Pipe Coated	Parameter	Initial Pipe Volume*	Epoxy Coating Volume	Material Thickness	Coating Surface Area to Water Volume Ratio
		mL	***cm^3***	***mm***	***cm^2/mL***
LSLs**	Maximum	306	73.7	1.21	3.01
	Minimum	228	31.7	0.52	2.46
	Mean	257	53.3	0.89	2.82
	SD	±25	±16	±0.28	±0.19
CSLs	Maximum	287	29.0	0.40	2.51
	Minimum	290	22.3	0.30	2.48
	Mean	289	24.2	0.36	2.49
	SD	±1.6	±2.3	±0.03	±0.01

* The initial pipe volumes of the CSLs are those of the first three CSLs measured and the mean value was assumed representative of all CSLs since the copper pipe was uniform, from the same coil, with each specimen cut to the same length.

** The nominal ID of the LSLs was 5/8 in. No pipes with a ½ in. nominal ID were used in the experiments described below.

Lead

Table 3 shows the lead concentrations in samples collected from fill-and-dump experiments using epoxy-coated pipe specimens. Lead concentrations in all extraction waters were below the detection level of 0.5 µg/L. The lead concentrations in the uncoated lead control pipe increased by 1,200 – 25,000 µg/L in the extraction waters, three orders of magnitude higher than values seen in 2011 in drinking water from the pipe's native distribution system in Rochester, N.Y. When collecting samples of extraction water from the uncoated control LSL, heavy white solids and lead-colored particles were visible. The white solids, which dissolved with the addition of HNO_3, could indicate the destablization of a lead-containing scale formed on the inner pipe wall. Scale dissolution after sample preservation undoubtedly contributed substantially to higher lead levels than would be seen if lead was only dissolving from the pipe walls. Furthermore, the pipes were heavily disturbed on both ends, where the pipes were reamed and threaded for SS nipple installation. Two heavy disturbances within only 4 feet of pipe length greatly increases the potential for lead spikes resulting from freshly exposed lead and detachment of lead particles. Short-term lead-level spikes in recently disturbed LSLs are consistent with published literature (Boyd *et al.* 2004; Sandvig *et al.* 2008), as described in Chapter 2. Spikes of the magnitude observed in this study would normally not be expected to occur in practice, but the heavily disturbed control samples used in this study provide an opportunity to evaluate the effectiveness of a lining or coating under conditions far more severe than those typically encountered in practice.

On average, lead concentrations in water samples drawn from the epoxy-coated LSLs were three orders of magnitude less than in samples from the uncoated control LSLs and one order of magnitude less than the AL of 15 µg/L. Figure 6 summarizes the lead concentrations in

samples collected from epoxy-coated pipe specimens. The median lead concentration in samples collected from epoxy-coated LSLs was below the MDL (0.5 µg/L). Since 75% of the samples collected from epoxy-coated CSLs contained no detectable amount of lead, the range represented in Figure 6 contains no box; it only contains the error bar representing the highest 25% of concentrations.

Table 2: Water quality parameters of extraction waters used in epoxy-coated pipe tests

Parameter	Units	pH 6.5 Extraction Water	Chlorinated pH 8.0 Extraction Water	Dechlorinated Tap Water	2011 Lawrence, Kan. Tap Water*	2012 Rochester, N.Y. Tap Water**
pH		6.5	8.0	8.4***	8.0 – 9.9	6.6 – 8.5
Alkalinity	*mg/L as $CaCO_3$*	1.8 – 100	56 – 100	126***	40 – 120	63 – 89
Sodium	*mg/L*			97	15 – 100	15 – 19
Potassium	*mg/L*			12	2.9 – 11	1.4 – 1.7
Chloride	*mg/L*				13 – 100	32 – 35
Sulfate	*mg/L*				27 – 150	13 – 30
Dissolved Oxygen	*mg/L*			8.2		
Conductivity	*µS/cm*			706	310 – 1,300	220 – 340
Hardness	*mg/L as $CaCO_3$*	100	100	96.5	100 – 190	91 – 130
Total Organic Carbon	*mg/L*	0.29 – 0.35	0.12 – 0.80	3.53	2.93 – 3.75	1.8 – 2.15
Copper	*µg/L*	≤ 0.25	≤ 0.25	5.7	6.3 – 120	12 – 320
Lead	*µg/L*	≤ 0.5	≤ 0.5	≤ 0.5	ND – 9.9	ND – 28

* 2011 Lawrence tap water values taken from the 2012 Lawrence Consumer Confidence Report, Appendix B (City of Lawrence 2012).
** 2012 Rochester, N.Y. tap water values taken from the 2012 Water Quality Report, Supplemental Information (City of Rochester 2012)
***pH and alkalinity measured prior to pH adjustment

Table 3: Lead concentrations in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Pb, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1,150	0.8
		6	Pb01	Cu01	78.3	≤ 0.5
		6	Pb02	Cu02	1.2	≤ 0.5
		24	Pb05	Cu05	3.8	0.6
		96	Pb07	Cu07	0.9	0.5
		240	Pb05	Cu05	0.8	0.7
	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb03	Cu03	≤ 0.5	≤ 0.5
		24	Pb06	Cu06	≤ 0.5	0.6
		96	Pb08	Cu08	1.2	≤ 0.5
		240	Pb06	Cu06	≤ 0.5	≤ 0.5
	pH 6.5 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1,800	0.6
		168	Pb04, Control	Cu10, Control	25,000	3.0
		6	Pb01	Cu01	≤ 0.5	≤ 0.5
		6	Pb02	Cu02	≤ 0.5	≤ 0.5
		168	Pb01	Cu01	≤ 0.5	≤ 0.5
		168	Pb02	Cu02	≤ 0.5	≤ 0.5
		6	Pb03	Cu03	≤ 0.5	≤ 0.5
		168	Pb03	Cu03	0.5	≤ 0.5
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1,700	≤ 0.5
		168	Pb04, Control	Cu10, Control	2,400	≤ 0.5
		6	Pb08	Cu08	≤ 0.5	≤ 0.5
		24	Pb02	Cu02	≤ 0.5	≤ 0.5
		24	Pb05	Cu05	2.11	≤ 0.5
		24	Pb09	Cu09	≤ 0.5	≤ 0.5
		168	Pb02	Cu02	≤ 0.5	≤ 0.5
		168	Pb05	Cu05	1.38	≤ 0.5
		168	Pb09	Cu09	≤ 0.5	≤ 0.5
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb02	Cu02		
		6	Pb05	Cu05	6.2	
		6	Pb08	Cu08		
		6	Pb09	Cu09	≤ 0.5	≤ 0.5
		24	Pb02	Cu02		
		24	Pb05	Cu05	2.0	
		24	Pb08	Cu08		
		24	Pb09	Cu09	≤ 0.5	≤ 0.5
		168	Pb02	Cu02		
		168	Pb05	Cu05	1.3	
		168	Pb08	Cu08		
		168	Pb09	Cu09		

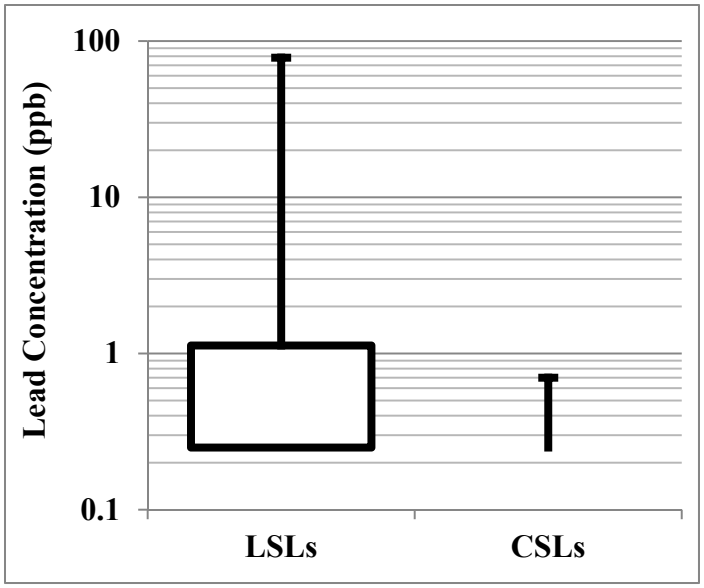

Figure 6: Lead concentrations in samples collected from epoxy-coated pipe specimens

Of the 27 samples collected from coated LSLs, only one had a lead concentration (78 µg/L) exceeding the AL of 15 µg/L. Another sample contained 6.2 µg/L Pb. The remaining 25 samples contained ≤ 3.8 µg/L Pb, which is within the range reported for Lawrence tap water (ND - 9.9 µg/L) in 2011. Lead concentrations in sixteen of the samples were at the detection level (0.5 µg/L) or lower. Of the 11 samples with a detectable lead level, 7 were collected from the same pipe specimen, Pb05, which contains a large joint. The pipe specimen that held the sample that contained 78 µg/L (Pb01) was reused twice to hold both pH 6.5 and chlorinated pH 8.0 extraction waters. In both cases, the lead levels measured were below the MDL. If the epoxy was unsuccessful at blocking lead release in pipe specimen Pb01, lead should have been detectable in subsequent tests. Furthermore, since lead is seen in significantly lower levels in all other samples from all other pipe specimens, it is reasonable to conclude that the 78 µg/L Pb measured in one sample was the result of contamination.

Figure 7 displays lead concentrations in samples collected from just the epoxy-coated LSLs (excluding the CSLs), separated into two groups: those exposed for the first time and those exposed in a previous test. Excluding the first sample collected from Pb01 (78 µg/L) as an outlier and all samples collected from Pb05 (discussed in more detail below), only 3 of remaining 6 samples collected from LSLs extracted for the first time contained detectable levels of lead, and the concentrations were quite low (1.2, 0.9, and 1.2 µg/L). These very low concentrations did not necessarily leach from or through the epoxy coating but could have resulted from surface contamination during the coating process that was not completely removed by flushing or from contamination introduced as the pipes and samples were handled in the laboratory. All 14 samples collected from previously exposed epoxy-coated LSLs, excluding samples collected from Pb05, contained lead below the MDL (0.5 µg/L). Thus, if leachable lead was initially present in any of the epoxy-coated LSLs, it was either removed by the 15-min. flushing or removed or passivated the first time the LSLs were extracted. Similar results would

presumably have been obtained using the protocols specified by NSF/ANSI Standard 61, which calls for the extraction water from the first two exposure periods to be discarded.

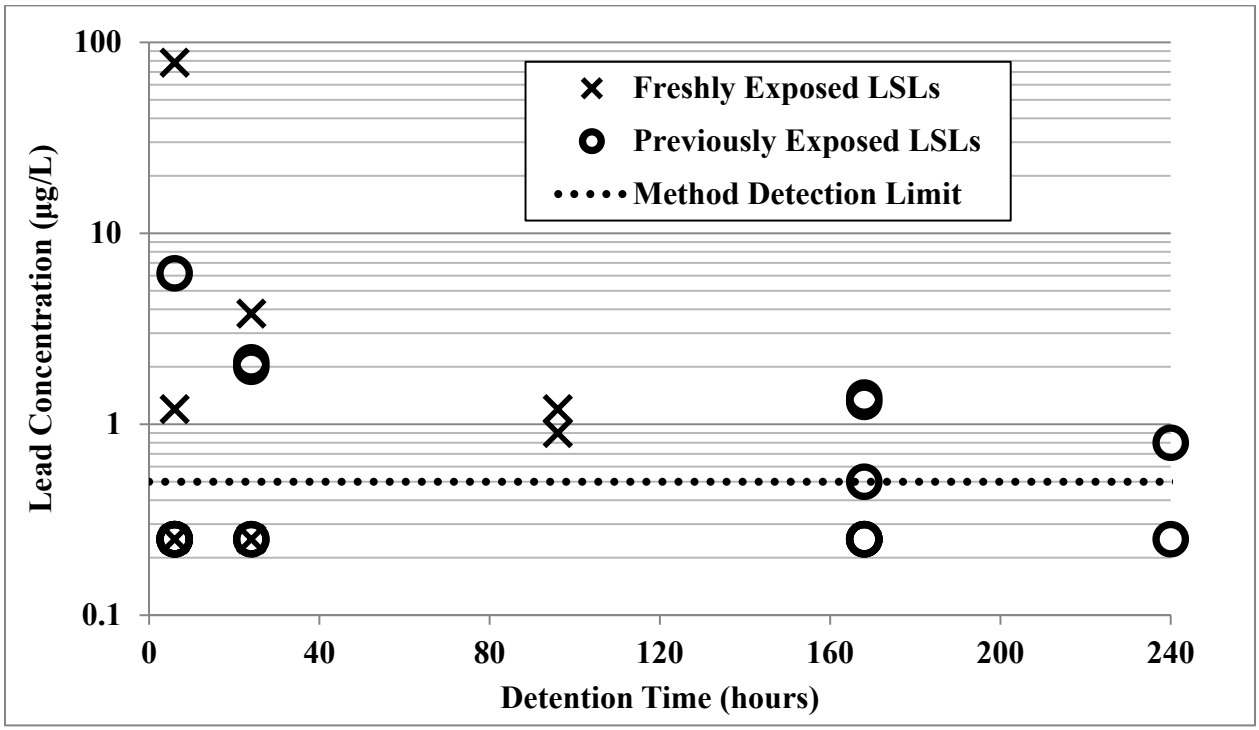

Figure 7: Lead concentrations in samples collected from coated LSLs

LSL specimen Pb05 is the only specimen that consistently produced samples containing detectable levels of lead (3.8, 0.8, 2.1, 1.4, 6.2, 2.0, and 1.3 µg/L), which is why this specimen was repeatedly sampled. As with all coatings, the application of epoxy carries a potential for defects. These defects can potentially propagate larger breaches in the coating surface, allowing water to contact lead surface and contribute to further corrosion of lead. There is a possibility that the epoxy never fully coated a portion of pipe Pb05, perhaps at the large solder joint (having a diameter approximately twice that of the rest of the pipe) located approximately half-way down its length. However, the pipe was carefully inspected with a pipe camera, both initially by the manufacturer (at the time the pipe was coated) and later by the author, and no defects were visible near the joint. A possible defect was visible (in the post-experiment inspection) at one end of the pipe where there appeared to be a very thin line of exposed metal that travelled the circumference of the SS nipple/lead pipe junction. This could explain the continued occurrences of detectable lead levels seen in samples collected from Pb05. Disturbing the SS nipple with enough shear force or torsion could compromise the epoxy in such a manner as to crack along the nipple/pipe junction. This could have occurred during the flushing procedure. In order to create a water-tight manifold to flush multiple pipe specimens at a time, female-to-female threaded fittings were installed to connect the SS nipples of two LSLs. Excessive twisting of the connecting fitting could have transferred enough torsion to the coated epoxy via the SS nipples to cause a crack. If the epoxy did not fully coat the inner pipe wall or was compromised, it was still effective at significantly reducing lead levels compared to the uncoated lead pipe. However,

if a small defect exists in the coating, it could pose long-term erosion and corrosion issues after continued exposure to flowing drinking water.

Copper

As shown earlier in Table 2, the dechlorinated tap water contained 5.7 μg/L copper while the pH 6.5 and chlorinated pH 8 extraction waters contained ≤ 0.25 μg/L copper. Table 4 displays copper concentrations in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens. All 51 extraction water samples collected from CSLs (n = 24) and LSLs (n = 27) contained a detectable level of copper (≥ 0.25 μg/L). Figure 8 summarizes the copper concentration increases in epoxy-coated pipe specimens. To determine copper increase, the copper concentrations in all dechlorinated tap water samples collected from epoxy-coated pipe specimens were corrected by subtracting the initial copper concentration (5.7 μg/L). In the uncoated control CSL, copper concentrations increased by 270 – 830 μg/L. Of the 51 extraction water samples exposed to coated pipe specimens, 48 samples increased in copper concentration compared to initial extraction water levels, but the increases were significantly less (1 – 2 orders of magnitude) than in extraction water exposed to the uncoated copper control pipe and approximately 2 orders of magnitude less than the AL for copper (1,300 μg/L).

Table 5 displays the mean copper concentration increases in samples collected from epoxy-coated pipes (CSLs and LSLs combined), separating data for samples collected from pipes exposed for the first time from the data for samples from previously exposed pipes. The mean increase in copper concentration decreased by approximately half in samples collected from previously exposed pipe specimens compared to samples collected from freshly exposed pipe specimens.

As discussed earlier, the epoxy contained approximately 8 mg copper/kg epoxy. Based on the epoxy density and applied volume of epoxy per pipe (Table 1), there was approximately 260 – 590 μg copper present in each pipe coating. Copper release from the epoxy may account for its presence in the samples. Another possibility could be contamination introduced when the freshly coated pipes were flushed with tap water, which contained approximately 5.7 μg/L copper. The copper may have adsorbed in small quantities to the epoxy during the flushing process and re-entered the extraction water during the experiments. Regardless of the mechanism, the amounts of copper seen in the epoxy-coated specimens are far less than the amounts seen in the uncoated control copper pipe and orders of magnitude less than the current AL for copper (1,300 μg/L). Furthermore, the range of copper increase seen in all samples from epoxy-coated pipes (0.4 – 22 μg/L) is within the range of copper concentrations observed in Lawrence, Kan. tap water in 2011 (6.3 – 120 μg/L).

Since copper concentrations decreased significantly (one-tailed t-test, $\alpha = 0.05$) after repeated fill-and-dump tests, as seen in Table 5 and Figure 9, copper may initially have been rapidly released from the surface, diminishing over time as the surface copper was exhausted. Copper release could continue, but more slowly, as it would be limited by its diffusion rate through the epoxy itself, which is presumed to be negligible. Since the highest copper concentrations found in samples from the epoxy-coated pipes were negligible relative to the AL

or copper, there appears to be no reason for utilities or consumers to be concerned about the copper present in the epoxy.

Table 4: Copper concentrations in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Cu, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		5.7	
		6	Pb04, Control	Cu10, Control	14	390
		6	Pb01	Cu01	15	15
		6	Pb02	Cu02	9.1	20.0
		24	Pb05	Cu05	9.3	7.6
		96	Pb07	Cu07	5.0	7.3
		240	Pb05	Cu05	5.5	3.1
	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb03	Cu03	6.8	7
		24	Pb06	Cu06	5.6	8.9
		96	Pb08	Cu08	10	15
		240	Pb06	Cu06	2.8	7.4
	pH 6.5 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb04, Control	Cu10, Control	5.5	830
		168	Pb04, Control	Cu10, Control	10	400
		6	Pb01	Cu01	2.6	6.3
		6	Pb02	Cu02	4.0	4.3
		168	Pb01	Cu01	7.1	15
		168	Pb02	Cu02	10	8.9
		6	Pb03	Cu03	2.1	1.9
		168	Pb03	Cu03	6.3	11
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb04, Control	Cu10, Control	3.7	270
		168	Pb04, Control	Cu10, Control	4.5	85
		6	Pb08	Cu08	2.7	3.3
		24	Pb02	Cu02	0.37	0.38
		24	Pb05	Cu05	2.6	1.3
		24	Pb09	Cu09	22	14
		168	Pb02	Cu02	1.1	1.0
		168	Pb05	Cu05	2.8	0.67
		168	Pb09	Cu09	4.1	3.8
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb02	Cu02		
		6	Pb05	Cu05	2.2	
		6	Pb08	Cu08		
		6	Pb09	Cu09	2.8	8.7
		24	Pb02	Cu02		
		24	Pb05	Cu05	1.6	
		24	Pb08	Cu08		
		24	Pb09	Cu09	1.8	2.7
		168	Pb02	Cu02		
		168	Pb05	Cu05	2.0	
		168	Pb08	Cu08		
		168	Pb09	Cu09		

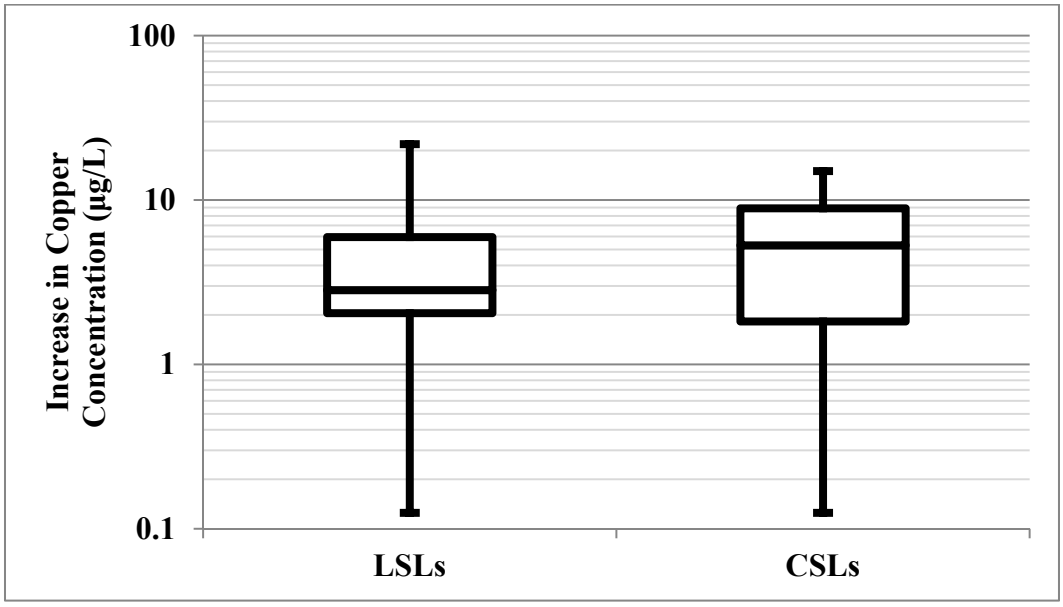

Figure 8: Increase in copper concentrations in samples collected from epoxy-coated pipe specimens

Table 5: Mean copper concentration increases in samples collected from epoxy-coated pipe specimens exposed to extraction water for the first time and previously exposed

Sample Subset	Mean Copper Concentration Increase (µg/L)	±SD (µg/L)	Number of Samples
All Samples	5.3	4.8	51
Freshly Exposed Pipes	8.3	5.8	16
Previously Exposed Pipes	3.9	3.5	35

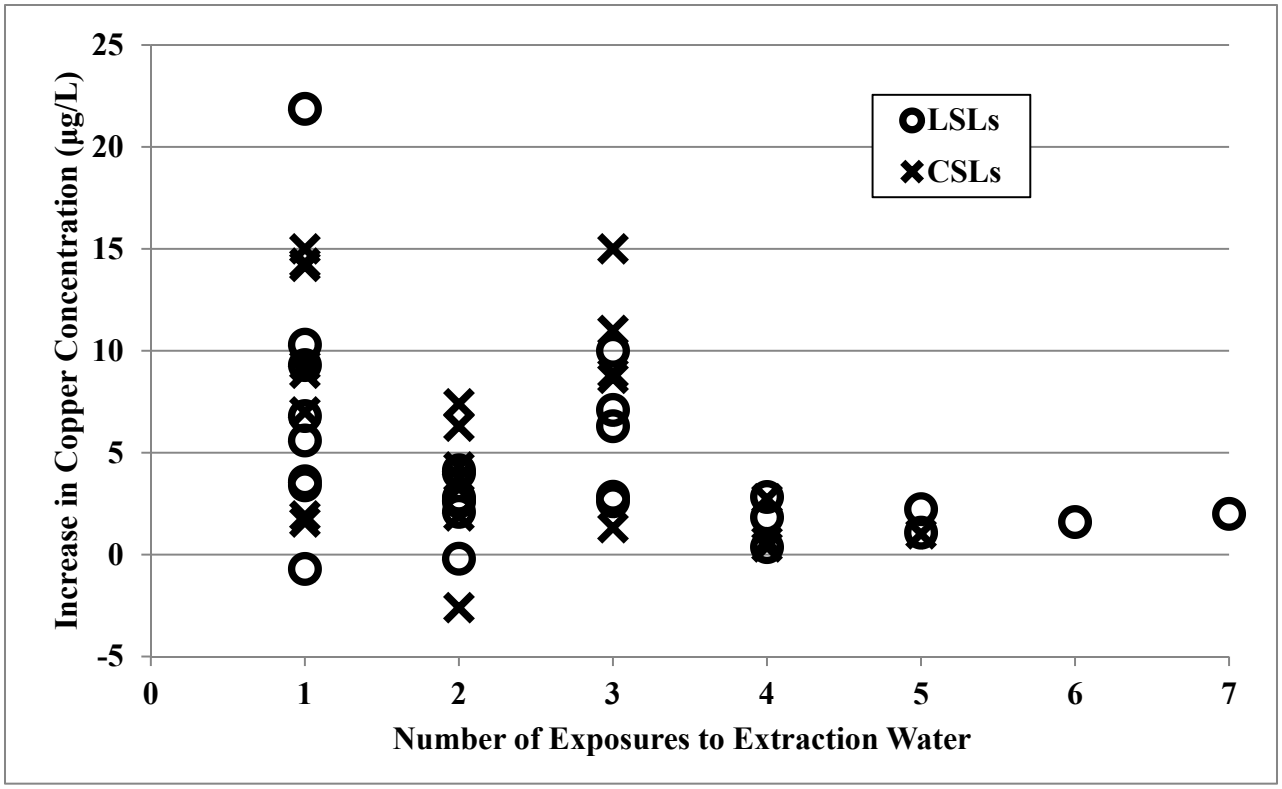

Figure 9: Increase in copper concentrations in epoxy-coated pipe specimens after sequential fill-and-dump tests

Total Organic Carbon

Table 6 displays the TOC concentrations in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens. Three values were omitted from statistical analysis performed for the discussion below: one for pipe specimen Pb08 (13.1 mg/L) when exposed to chlorinated pH 8 water for 4 days, and two for pipe specimen Cu02 (4.78 mg/L and 4.36 mg/L) when exposed to dechlorinated tap water for 6 hours and pH 6.5 extraction water for 7 days, respectively. All three values were determined to be statistical outliers (Grubb's, $\alpha = 0.05$). Other values with excessively high %RSDs (3.06 ± 4.2%, 1.38 mg/L ± 6.3%, 0.78 mg/L ± 5.4%, and 0.51 mg/L ± 11.3%) were included in data analysis because they were not statistical outliers (Grubb's test, $\alpha = 0.05$) and the results from sequential injections were not trending strongly enough to suggest that the TOC analyzer was experiencing a membrane equilibration issue.

As displayed in Figure 10 and Table 7, the average initial TOC concentrations in the dechlorinated tap water, chlorinated pH 8.0 extraction water, and pH 6.5 extraction water were 3.53 mg/L, 0.48 mg/L, and 0.32 mg/L, respectively. Samples collected from epoxy-coated pipe specimens exposed to dechlorinated tap water, chlorinated pH 8.0 extraction water, and pH 6.5 extraction waters contained mean TOC concentrations of 3.48 ± 0.27 mg/L ($n = 9$), 0.87 ± 0.50 mg/L ($n = 33$), and 1.19 ± 0.79 mg/L ($n = 11$) for mean increases of -0.05 mg/L, +0.39 mg/L, and +0.87 mg/L, respectively. The difference in TOC concentration before and after exposure to the epoxy was not statistically significant for the dechlorinated tap water (two-tailed t-test, $\alpha = 0.05$) but the increase was statistically significant (one-tailed t-test, $\alpha = 0.05$) for the chlorinated pH 8 extraction water and the pH 6.5 extraction water. The mean increase in TOC concentration for all samples of the latter two extraction waters exposed to freshly epoxy-coated pipes was 0.65 ± 0.62 mg/L ($n = 44$). This slight increase is consistent with previous research (Heim and Dietrich 2007).

As noted in Chapter 2, some components of TOC in drinking water, such as humic and fulvic acids, can complex with copper and might help draw it into solution. Furthermore, the epoxy appeared to be capable of leaching both TOC and copper, and at least one typical epoxy component (polyamines) can potentially bind copper and might therefore help draw it into solution. Therefore, the data were examined to determine if the increases in TOC and copper were correlated. Figure 11 displays the relationship between TOC and copper concentration increases in all samples collected from epoxy-coated pipe specimens. The relationship between TOC and copper concentrations was not significant ($\alpha = 0.05$) in samples collected from either CSLs or LSLs. Thus, the data do not suggest a close relationship between TOC release and copper release.

Table 6: TOC concentrations in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		TOC, mg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		3.53	
		6	Pb04, Control	Cu10, Control	3.62	3.62
		6	Pb01	Cu01	3.28	3.46
		6	Pb02	Cu02	3.36	4.78**
		24	Pb05	Cu05	3.65	3.48
		96	Pb07	Cu07	3.76	3.99
		240	Pb05	Cu05	3.18	3.19
	Chlorinated Extraction Water, pH 8		Extraction Water		0.80/0.45*	
		6	Pb03	Cu03	0.96	1.36
		24	Pb06	Cu06	0.92	1.10
		96	Pb08	Cu08	13.10**	1.72
		240	Pb06	Cu06	0.77	1.11
	pH 6.5 Extraction Water		Extraction Water		0.35/0/29*	
		6	Pb04, Control	Cu10, Control	0.50	0.31
		168	Pb04, Control	Cu10, Control	1.00	0.28
		6	Pb01	Cu01	0.52	1.38 ± 6.3%†
		6	Pb02	Cu02	0.73	1.48
		168	Pb01	Cu01	0.78 ± 5.4%†	3.06 ± 4.2%†
		168	Pb02	Cu02	1.68	4.36 ± 2.8%†**
		6	Pb03	Cu03	0.39	0.86
		168	Pb03	Cu03	0.49	1.71
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		0.19/0.20*	
		6	Pb04, Control	Cu10, Control	1.87	0.41
		168	Pb04, Control	Cu10, Control	1.02	0.25
		6	Pb08	Cu08	1.46	0.91
		24	Pb02	Cu02	0.36	0.34
		24	Pb05	Cu05	0.81	1.01
		24	Pb09	Cu09	0.95	2.66
		168	Pb02	Cu02	0.51	0.54
		168	Pb05	Cu05	0.58	0.80
		168	Pb09	Cu09	0.88	1.46
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		0.51 ± 11.3%†/0.17*	
		6	Pb02	Cu02		
		6	Pb05	Cu05		
		6	Pb08	Cu08		
		6	Pb09	Cu09	0.49	0.43
		24	Pb02	Cu02		
		24	Pb05	Cu05		
		24	Pb08	Cu08		
		24	Pb09	Cu09	0.46	0.46
		168	Pb02	Cu02	0.44	0.43
		168	Pb05	Cu05	0.48	0.52
		168	Pb08	Cu08	1.75	0.73
		168	Pb09	Cu09	0.69	0.71

* The data points represent extraction water used for a detention time of 6 – 24 hours and 7 days, respectively
** Data value omitted from data analysis as an outlier
† %RSD values are shown for data points that had excessive %RSD based on criteria outlined in Chapter 3.

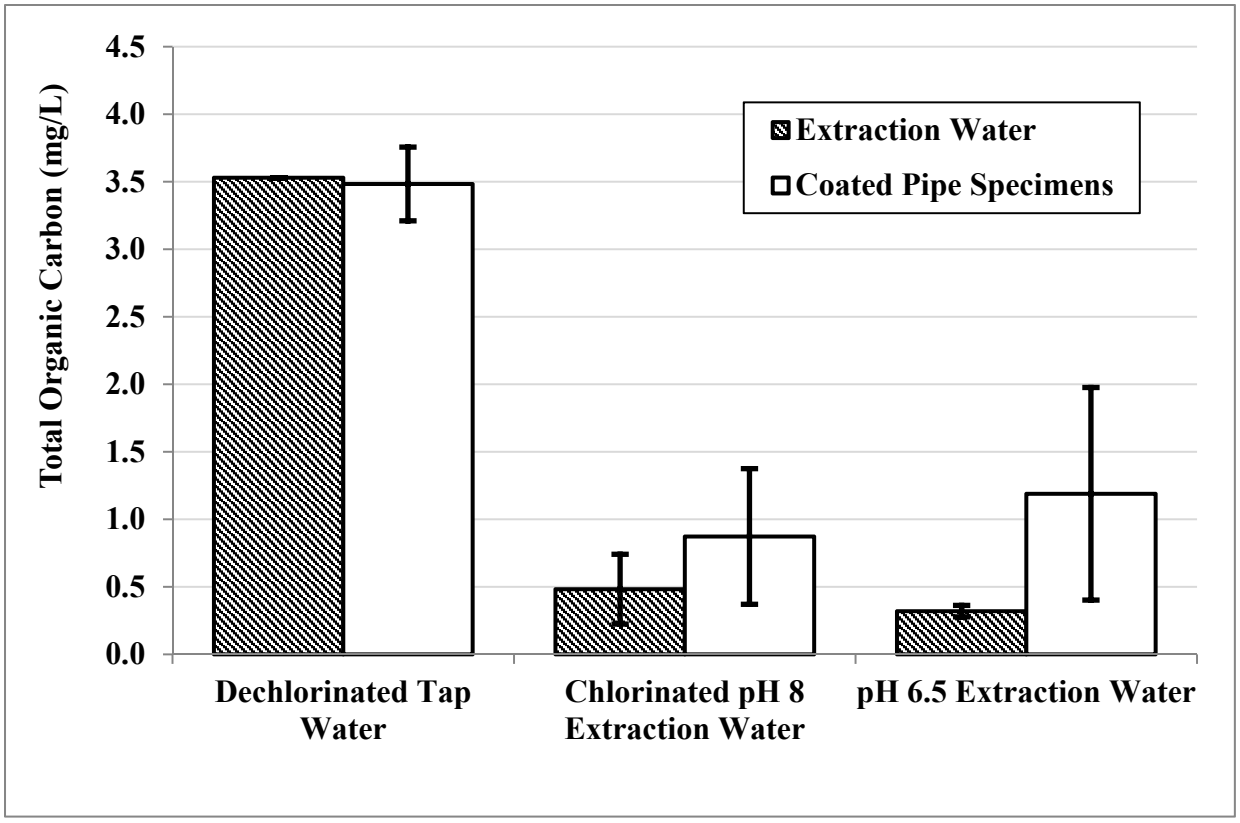

Figure 10: TOC concentrations in samples collected from epoxy-coated pipe specimens and reservoirs (Error bars represent SD)

Table 7: Mean TOC concentrations in initial extraction waters and in samples collected from epoxy-coated pipes

Extraction Water	Sample Subset	Mean TOC Concentration (mg/L)	±SD (mg/L)	Number of Samples
Dechlorinated Tap Water	Initial	3.53	N/A	1
	Exposed to Coated Pipe Specimen	3.48	0.27	9
Chlorinated pH 8 Extraction Water	Initial	0.48	0.26	4
	Exposed to Coated Pipe Specimen	0.87	0.50	33
pH 6.5 Extraction Water	Initial	0.32	0.04	2
	Exposed to Coated Pipe Specimen	1.19	0.79	11

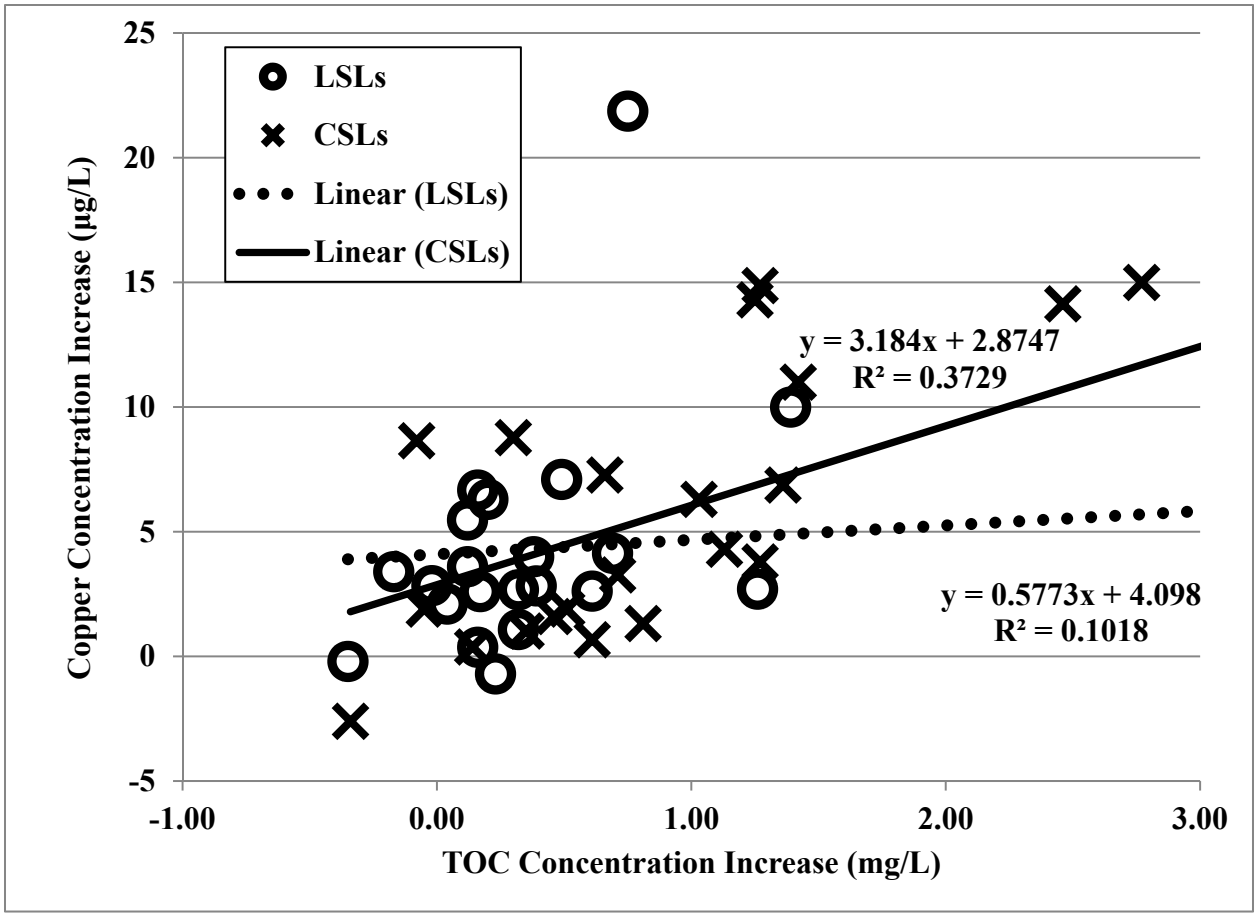

Figure 11: Relationship between TOC concentration increase and copper concentration increase in samples collected from epoxy-coated pipe specimens

Residual Chlorine

All chlorinated pH 8 extraction water samples exposed to epoxy-coated pipes were analyzed for total chlorine. Monochloramine was measured in some samples initially containing free chlorine, to verify that the total chlorine measured was free chlorine and not combined chlorine formed by the reaction of chlorine with the epoxy, which contains amines. All measured monochloramine concentrations in samples exposed to free chlorine were ≤ 8% of the total chlorine measured; therefore, it is assumed in the following discussion that any total chlorine measured was free chlorine unless the samples were initially chloraminated. The small amounts of combined chlorine detected in samples exposed to free chlorine may have resulted from "carryover" (due to reactions of free chlorine with the reagents for monochloramine analysis) or organic chloramines formed by reaction of chlorine with amines present in the epoxy.

Chlorine demand was determined by subtracting the chlorine residual measured in samples from the initial chlorine residual in the extraction water. The free chlorine remaining in samples from the uncoated control pipes can be viewed in Figure 12, the data for which can be found in Table 22 in Appendix D. Figure 13 displays the free chlorine remaining in samples

from the epoxy-coated pipe specimens, the data for which can be found in Table 23 in Appendix D. The coated pipes consumed nearly all the free chlorine after 6 hours. The uncoated control pipes were not exposed to free chlorine for a 6 hour detention time, but consumed nearly all the free chlorine after 24 hours.

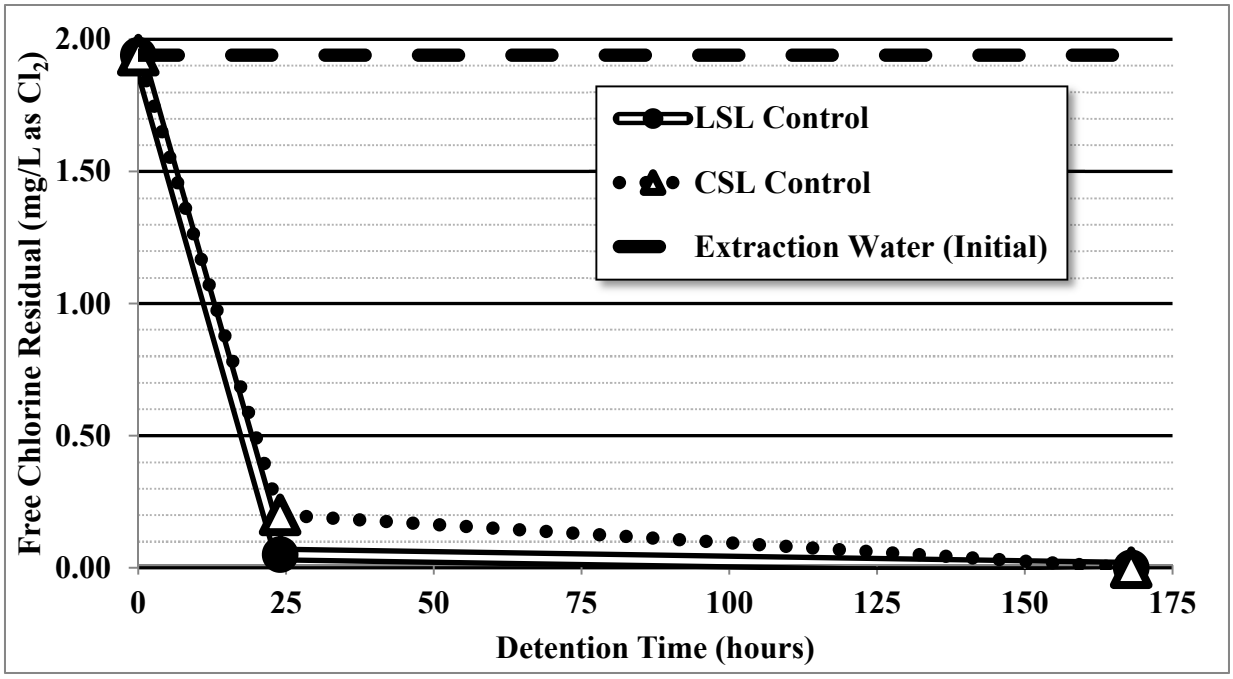

Figure 12: Free chlorine remaining in uncoated control pipes over time after exposure to chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of 1.94 mg/L free chlorine as Cl_2

Remaining monochloramine concentrations in samples collected from epoxy-coated pipe specimens are displayed in Figure 14, the data for which can be found in Table 24 in Appendix D. The monochloramine concentration in the control sample (chlorinated pH 8 extraction water stored in a 4 L amber glass bottle) decreased 0.34 mg/L as Cl_2 after 4 days. The epoxy-coated pipes consume chlorine at a much faster rate, 1.3 mg/L as Cl_2 and 1.6 mg/L as Cl_2 after 6 hours in the epoxy-coated LSLs and CSLs, respectively. Comparatively, the uncoated control LSL consumed only 0.4 mg/L as Cl_2 after 6 hours. The uncoated control CSL consumed approximately the same amount of monochloramine as the coated CSL after 6 hours, 1.5 mg/L as Cl_2. The epoxy-coated pipe specimens' combined chlorine demand was much like their free chlorine demand, with nearly all the combined chlorine being consumed after 24 hours. The results indicate a reactive epoxy surface that initially consumes substantial amounts of chlorine.

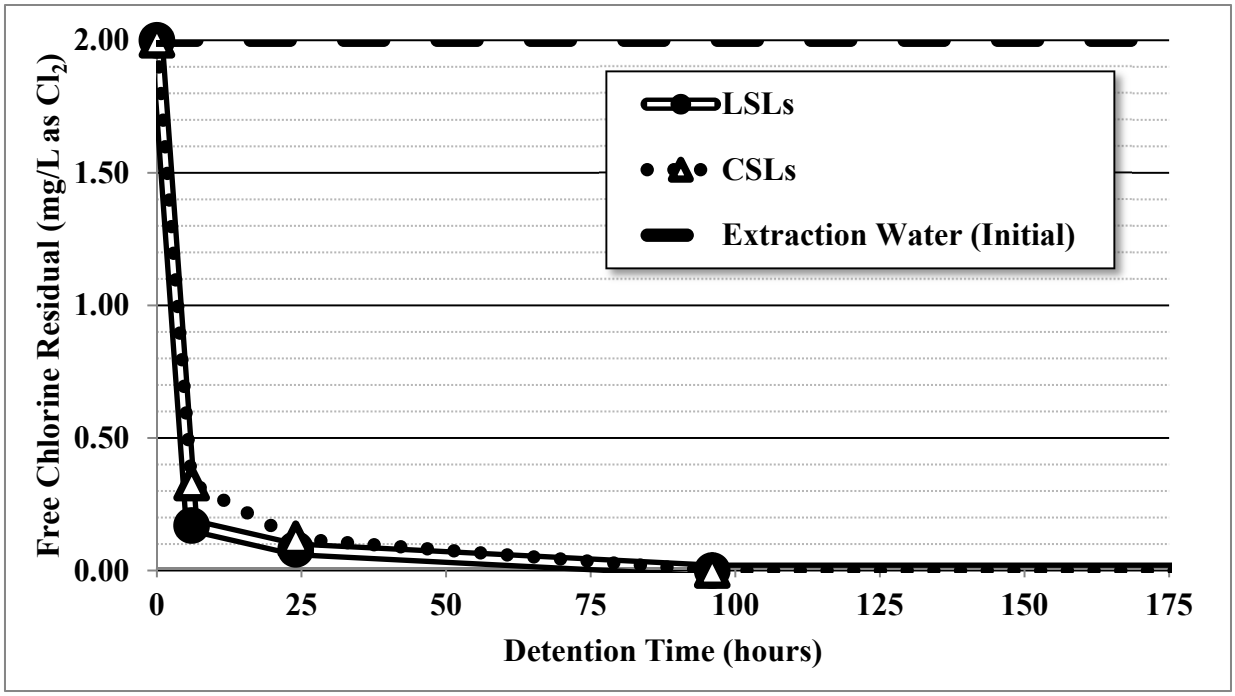

Figure 13: Free chlorine remaining in epoxy-coated control pipes over time after exposure to chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of 2.00 mg/L free chlorine as Cl_2

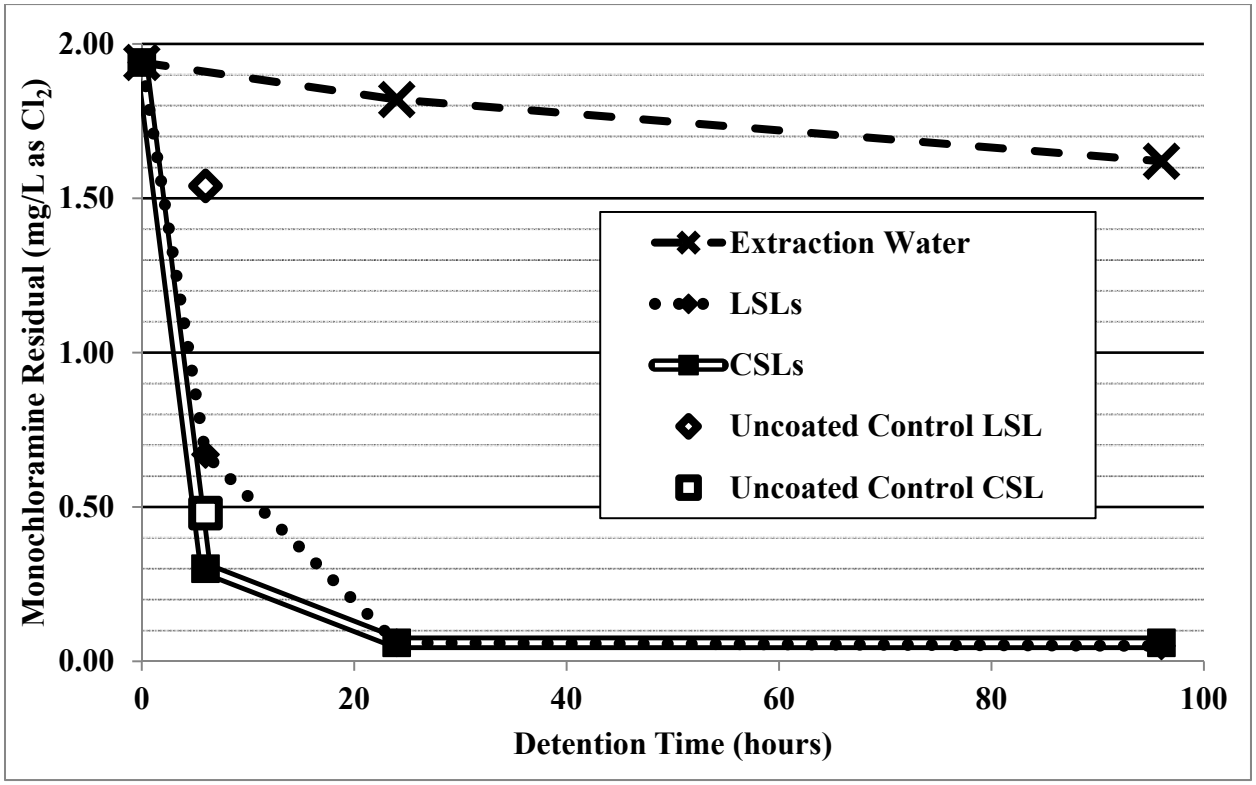

Figure 14: Monochloramine remaining in samples collected from epoxy-coated pipe specimens over time after exposure to pH 8.0 extraction water containing 1.94 mg/L combined chlorine as Cl_2

In a series of 1-hour fill-and-dump tests sequentially exposing the same epoxy-coated LSL and CSL specimens (Pb06 and Cu06, respectively) to chlorinated pH 8 extraction water, the remaining free chlorine gradually increased with the number of tests conducted, as seen in Figure 15 (the values for which are listed in Table 25 in Appendix D). After nine 1-hour tests with 2 mg/L free chlorine as Cl_2, two 3-hour tests were performed with 100 mg/L free chlorine as Cl_2 (Figure 16, the data for which are shown in Table 26 in Appendix D). The pipes were then subjected to 6 more sequential 1-hour tests with chlorinated pH 8 extraction water dosed with 2 mg/L as Cl_2 and then 3 more sequential 3-hour tests exposed to 100 mg/L as Cl_2. After repeated exposure, the free chlorine demand stabilized at approximately 10% of the initial free chlorine concentration when exposed to either 2 mg/L as Cl_2 for 1 hour or 100 mg/L as Cl_2 for 3 hours.

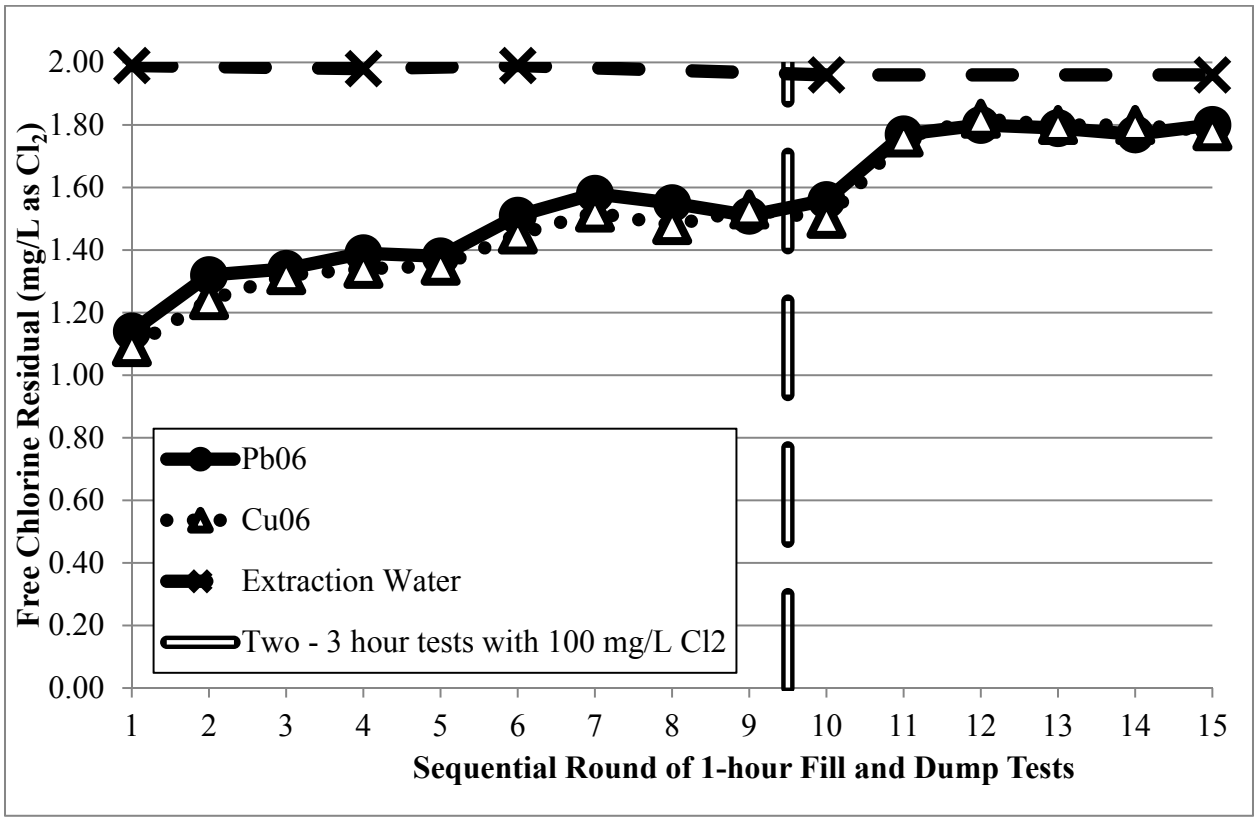

Figure 15: Free chlorine remaining after sequential 1-hour fill-and-dump experiments with chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of 2.00 mg/L free chlorine as Cl_2

After repeated exposure, the number of active sites on the epoxy surface should be decreasing. If chlorination of polyamides is occurring, chlorine could be bonding to the surface and taking up these active sites over time. However, since the chlorine demand was still substantial (though leveling off) over time, it appears that the chlorine is not reacting only with the outer surface of the epoxy coating. It may be diffusing into the coating and reacting with amines or polyamides, or it may be reacting with compounds leaching up through the epoxy coating. It is also conceivable that the chlorine is simply diffusing though the epoxy coatings and reacting directly with the pipe walls. If any iron existing in the iron oxide pigment used in

the epoxy is in a reduced form, such as Fe(II), it might readily react with chlorine, which could also explain the observed chlorine demand.

The significant chlorine demand of the epoxy coating creates the potential for the formation of DBPs. Efforts to identify and quantify DBPs were beyond the scope of this study, but are being made as part of a companion study.

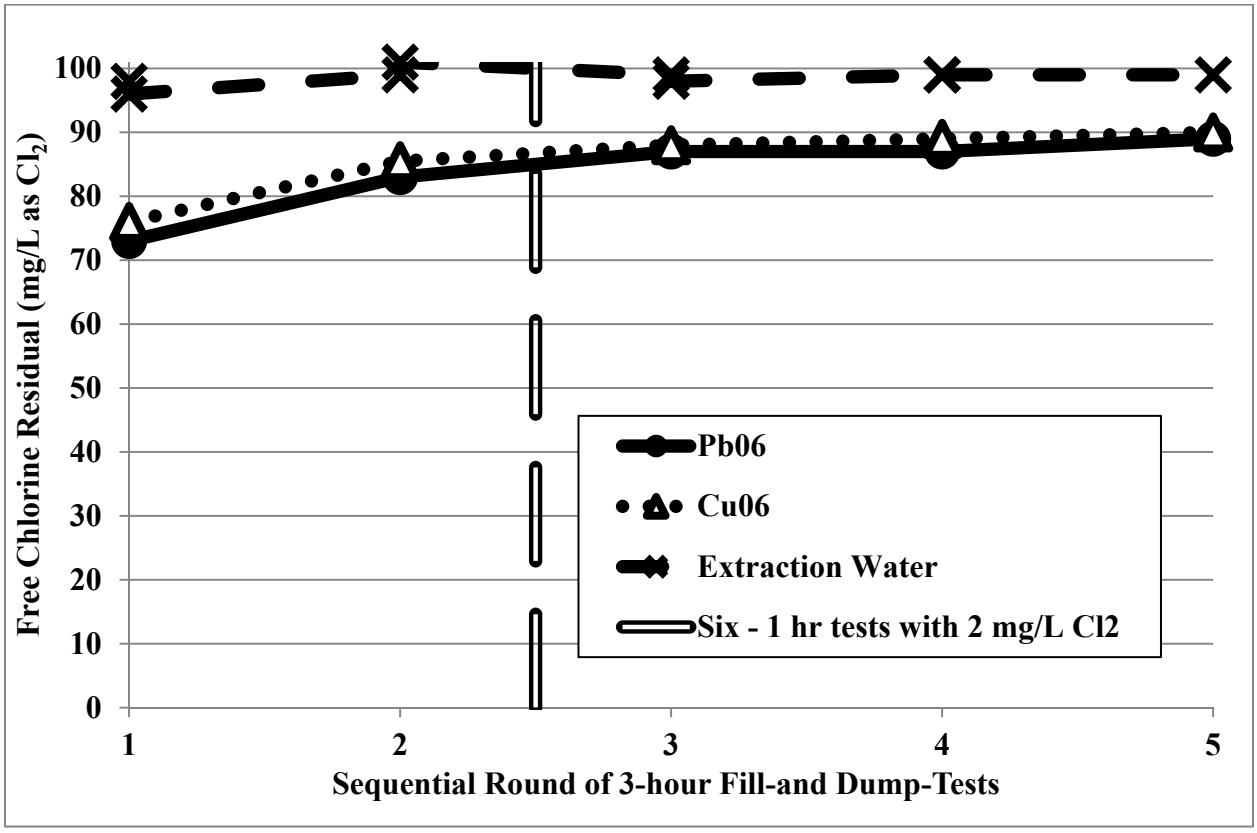

Figure 16: Free chlorine remaining after sequential 3-hour fill-and-dump experiments with chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of 100 mg/L free chlorine as Cl_2

PET-LINED PIPE SPECIMENS

Elemental Analysis of PET Liner

Analysis of the PET liner using ICP-MS and ablating the liner surface with a laser indicated the presence of antimony, as shown in Figure 17. However, the amount of antimony was not quantifiable using the ablation technique since the amount of liner drawn from the solid by the laser is itself unknown. Therefore, a digestion was performed on a known mass of liner to provide a sample from which could produce quantitative results. ICP-MS analysis quantified the average antimony concentration in the PET liner at 130 mg Sb/kg PET. This result is only slightly below the range of 190 – 300 mg/kg reported by Duh (2002), and represents a quantity that could potentially pose health risks if extensive leaching into drinking water were to occur.

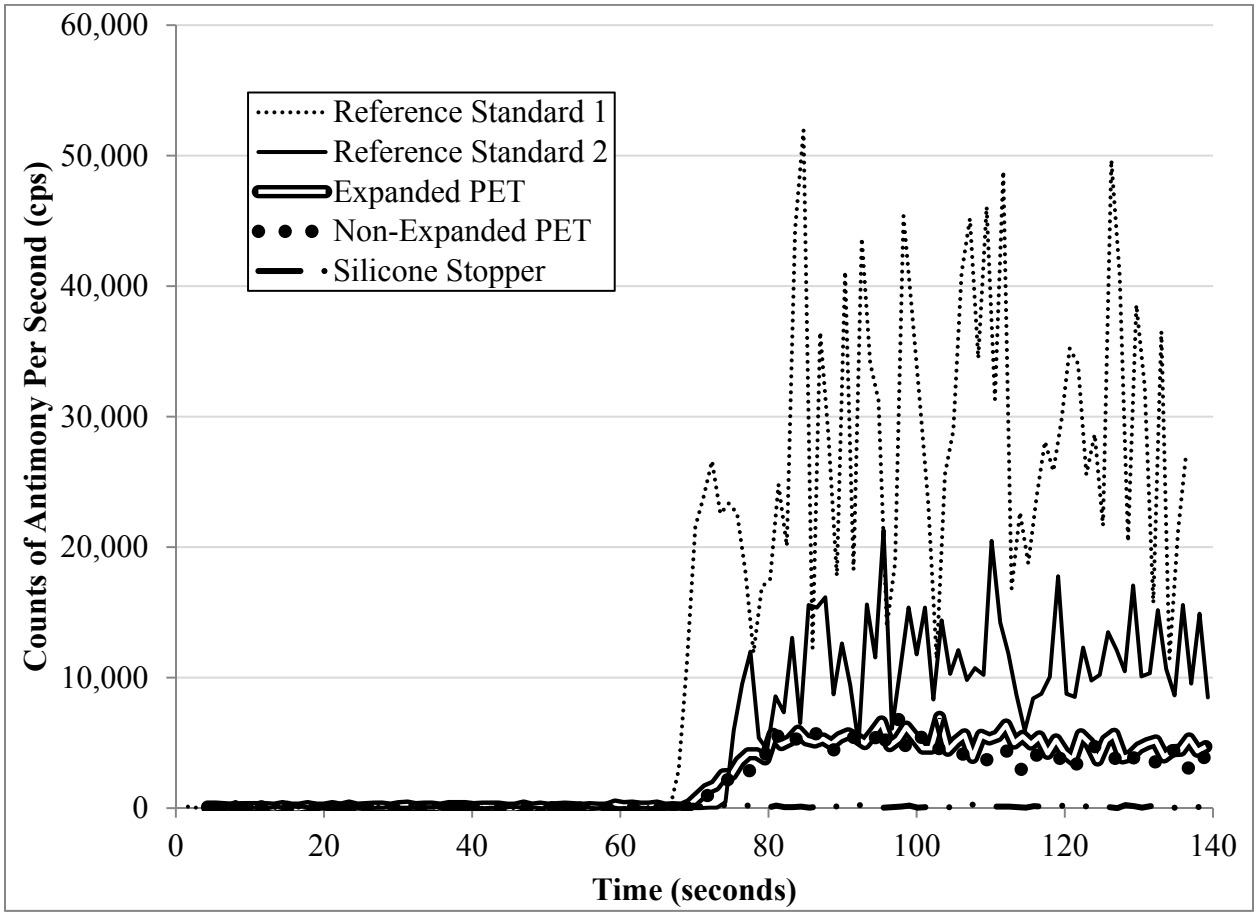

Figure 17: ICP-MS results for antimony in PET liner and silicone stopper using laser ablation

ICP-MS analysis of digested PET liner solutions also indicated the presence of chromium, which is inconsistent with the ICP-MS results using laser ablation. When testing lower-molecular-weight elements, the ICP-MS technique has the potential to pick up compounds of similar molecular weight. In this case, ClO^- present in the plasma (formed by oxidation of chloride in the sample) could be mistaken as chromium. To confirm the ICP-MS results, the

same samples were tested for chromium using Graphite Furnace Atomic Absorbance Spectrometry (GFAAS). The GFAAS technique should not confuse similarly weighted compounds for chromium, as this analysis is based on light absorbance at an element-specific wavelength. The results yielded chromium concentrations below the MDL of 0.5 mg Cr/kg PET, over two orders of magnitude less than the ICP-MS results of 90 mg Cr/kg PET. The results of the ICP-MS ablation and GFAAS tests both indicate that there was no measureable amount of chromium in the PET liner, and that the results of the quantitative ICP-MS test were therefore due to interference.

Physical PET Liner Characteristics

Measurements were taken of the pipe specimens, with the end fittings in place, before and after PET liner installation, to determine physical characteristics of the pipe and liner, as shown in Table 8. Since the CSL volumes were higher than the LSL volumes, the CSL SA:V ratios were actually lower than LSL SA:V ratios. The average inner diameter of lined CSLs was slightly larger than that of lined LSLs; 1.58 ± 0.01 cm versus 1.48 ± 0.05 cm, respectively.

Table 8: Physical PET liner and pipe characteristics

	Parameter	Initial Pipe Volume*	Lining Volume	Lining Thickness	Lining Surface Area to Water Volume Ratio
		mL	***cm^3***	***mm***	***cm^2/mL***
LSLs	Maximum	276	37.9	0.61	2.8
	Minimum	239	32.8	0.50	2.6
	Mean	259	35.3	0.56	2.7
	SD	±12.9	±1.7	±0.03	±0.09
CSLs	Maximum	294	39.6	0.59	2.5
	Minimum	291	33.8	0.50	2.5
	Mean	292	37.6	0.56	2.5
	SD	±2.0	±1.8	±0.03	±0.01

* The initial pipe volumes of the CSLs are those of the first three CSLs measured and the mean value was assumed representative of all CSLs since the copper pipe was uniform, from the same coil, with each specimen cut to the same length.

Extraction Waters

As seen in Table 9, the water quality parameters for the dechlorinated tap water were within the ranges reported in 2011 for Lawrence, Kan. tap water, except for alkalinity. However, considering the variance in surface water quality from year to year (and season to season), the alkalinity measured in the dechlorinated tap water was not unreasonable. All values for dechlorinated tap water measurements listed in Table 9 were measured after dechlorination and pH adjustment, except pH and alkalinity. The pH was adjusted to 8.0 and the alkalinity was lowered by the addition of 0.1 M HCl.

Table 9: Water quality parameters of extraction waters used in PET-lined pipe tests

Parameter	Units	pH 6.5 Extraction Water	Chlorinated pH 8.0 Extraction Water	Dechlorinated Tap Water	2011 Lawrence, Kan. Tap Water*	2012 Rochester, N.Y. Tap Water**
pH		6.5	8.0	8.3***	8.0 – 9.9	6.6 – 8.5
Alkalinity	*mg/L as $CaCO_3$*	1.8	56	122***	40 – 120	63 – 89
Sodium	*mg/L*			18	15 – 100	15 – 19
Potassium	*mg/L*			3.8	2.9 – 11	1.4 – 1.7
Chloride	*mg/L*				13 – 100	32 – 35
Sulfate	*mg/L*				27 – 150	13 – 30
Dissolved Oxygen	*mg/L*	N/A	N/A	8.5	N/A	N/A
Conductivity	*µS/cm*			390	310 – 1,300	220 – 340
Hardness	*mg/L as $CaCO_3$*	100	100	118	100 – 190	91 – 130
Total Organic Carbon	*mg/L*	0.08	0.15	3.34	2.93 – 3.75	1.8 – 2.15
Copper	*µg/L*	≤ 0.25	≤ 0.25	23	6.3 – 120	12 – 320
Lead	*µg/L*	≤ 0.5	≤ 0.5	≤ 0.5	ND – 9.9	ND – 28
Antimony	*µg/L*	≤ 0.06	≤ 0.06	0.19	N/A	ND

* 2011 Lawrence tap water values taken from the 2012 Lawrence Consumer Confidence Report, Appendix B (City of Lawrence 2012).
** 2012 Rochester, N.Y. tap water values taken from the 2012 Water Quality Report, Supplemental Information (City of Rochester 2012)
***pH and alkalinity measured before pH adjustment

Lead and Copper

As shown in Table 9, lead and copper concentrations in pH 6.5 and pH 8.0 chlorinated extraction waters were non-detectable (≤ 0.5 μg/L Pb and ≤ 0.25 μg/L Cu, respectively). The lead concentration in the dechlorinated tap water was also below the MDL, while the copper concentration (23 μg/L) was within the levels seen in 2011 in Lawrence, Kan. drinking water (6.3 – 120 μg/L). Lead concentrations increased in the unlined control LSL by 1,400 – 21,000 μg/L. Copper concentrations in the unlined CSL control increased by 310 – 910 μg/L.

As mentioned in Chapter 3, water infiltrated the space between the liner pipe wall during the flushing procedure, but the significance of this was not immediately recognized. The fill-and-dump experiments proceeded as planned with additional tests performed after removing the SS nipples and cleaning the ends of the PET liners. After analyzing the samples and finding that many of them contained detectable levels of lead and copper, and then noting the very high concentrations of lead and copper found in the unlined (control) pipe specimens, it was recognized that the lead and copper results had very likely been compromised by contamination from water that had infiltrated behind the liners and then passed into the samples, perhaps by means of capillary action. Based on lead concentrations seen in unlined control LSLs, a volume on the order of only 10 – 1,000 μL of infiltrated would be needed to obtain the lead levels measured in samples collected from PET-lined LSLs. As the SS nipples were removed, droplets of water were observed on the newly exposed PET liner, resting between the liners and SS nipples. The volume of water in these droplets alone would be enough to contaminate the PET-lined LSL samples to their measured lead concentrations. Each end of each pipe was tightly stoppered during the fill-and-dump experiments, isolating the extraction water from the water resting between the PET liner and pipe wall. Therefore, if contamination did occur, it likely happened while pouring the extraction water from the pipe specimens into a glass beaker for analysis.

After investigation and discussions with the manufacturer, it became clear that the end fittings used in these experiments (the SS nipples) were incompatible with proper installation of PET liners. With SS nipples, the liner and pipe wall are parallel throughout the length of the pipe with no barrier to seal the liner. When water is flowing through the pipe, as was the case during the flushing procedure, there is nothing keeping the water from flowing behind the liner. When liners are installed in the field, a flanged end fitting is used, and the expanded liner is molded onto it (Figure 18), creating a physical barrier to stop water from flowing between the liner and pipe wall. Therefore, any lead and copper seen in the samples collected from the PET-lined pipe specimens could simply have circumvented the liner, so the results cannot be used as a measure of the PET liner's effectiveness to block lead and copper leaching. Therefore, the lead and copper data were deemed to have been compromised and were discarded. They will be replaced (in the on-going project this work is one part of) with data obtained using PET-lined specimens having proper end fittings that do not allow water to pass behind the liners.

Figure 18: Image depicting the difference in PET-lined pipes with: SS nipple used in the fill-and-dump experiments (top) and flanged fitting properly designed to seal liner after expansion (bottom)

Effective and efficient lining and coating methods have been carefully developed by manufacturers and installers based on many years of practical experience and research. Their installation procedures (summarized in the Background and Methodology sections) clearly place a high degree of importance on properly preparing a pipe before inserting a liner or applying a coating. The failure in this study to prevent water from passing behind the lining reinforces the importance, already recognized by manufacturers and installers, of properly preparing a pipe for lining or coating.

Antimony

Table 10 and Figure 19 display antimony concentrations in samples collected from fill-and-dump experiments on PET-lined pipe specimens. Dechlorinated tap water contained 0.19 µg/L antimony, while the pH 6.5 and chlorinated pH 8 extraction waters each contained no detectable level of antimony (≤ 0.06 µg/L). All but two of the samples collected from PET-lined LSLs and every sample collected from a PET-lined CSL contained detectable antimony (>0.06 µg/L), although the measured concentrations were all quite low. As shown in Table 11, the average level of antimony in dechlorinated tap water, chlorinated pH 8 extraction water, and pH 6.5 extraction water samples collected from PET-lined pipes were 0.28 ± 0.04 µg/L (n = 8), 0.09 ± 0.06 µg/L (n = 6), and 0.29 ± 0.18 µg/L (n = 12), respectively. The mean level of antimony in each subset of extraction water samples (dechlorinated tap, chlorinated pH 8, and pH 6.5) exposed to PET-lined pipes was statistically greater than the initial antimony levels present in the corresponding control sample of extraction water (one-tailed t-test, $\alpha = 0.05$).

The antimony increase in each of the 26 samples collected from PET-lined pipe specimens (13 LSLs and 13 CSLs) was determined by subtracting the antimony level initially present in the corresponding extraction water. Table 29 in Appendix E lists the antimony concentration increases in extraction water samples exposed to PET-lined pipe specimens. The antimony increases in the PET-lined pipe specimens were statistically significant (one-tailed t-test, $\alpha = 0.05$) for each extraction water, with a mean antimony increase of 0.18 ± 0.16 µg/L for all 26 samples. In comparison, the antimony concentration increases in samples collected from the unlined (control) LSL and CSL were 0.42 – 3.94 µg/L and 0.00 – 0.13 µg/L, respectively.

The increases in antimony concentration in samples collected from PET-lined pipes are summarized in Figure 19, which compares the data from the LSLs to that of the CSLs. The median antimony increase in the samples exposed to PET-lined LSLs was approximately the same as the median increase in samples collected from PET-lined CSLs (0.13 µg/L for both). In fact, both the maximum and minimum antimony levels for the samples collected from PET-lined CSLs were higher than those for samples collected from PET-lined LSL, as were the 25th and 75th percentiles. If SA:V values were higher for lined CSLs than for lined LSLs, this could explain the slightly higher antimony levels from lined CSLs, since a higher surface area per unit volume would allow more antimony to diffuse into a smaller volume water of water. However, the SA:V values are actually slightly lower for lined CSLs than for lined LSLs (Table 8).

As noted earlier, antimony leaching from PET water bottles increases with time (Cheng *et al.* 2010; Reimann, Birke, and Filzmoser 2010). A similar relationship can be seen in Figure 20, which compares the increases seen in samples exposed for shorter times (6 – 24 hours) to those exposed for longer times (4 days). The antimony increase in extraction water samples exposed to PET-lined pipe specimens for 6 – 24 hours was statistically significant (one-tailed t-test, $\alpha = 0.05$) with a mean of 0.09 ± 0.04 µg/L (n = 16). The antimony increase in with a mean 0.33 ± 0.17 µg/L (n = 10). The mean antimony increase in extraction water samples exposed to PET-lined pipe specimens exposed for 4 days was statistically greater than samples exposed for 6 – 24 hours (one-tailed t-test, $\alpha = 0.05$).

Table 10: Antimony concentrations in samples collected from fill-and-dump experiments on PET-lined pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Sb, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 3 (FD-03)	Dechlorinated Tap Water, pH 8	0	Extraction Water		0.19	
		6	Pb11, Control	Cu11, Control	4.13	0.19
		6	Pb12	Cu12	0.24	0.31
		6	Pb13	Cu13	0.24	0.28
		24	Pb15	Cu15	0.25	0.25
		96	Pb17	Cu17	0.34	0.34
	Chlorinated pH 8 Extraction Water	0	Extraction Water		≤ 0.06	
		6	Pb14	Cu14	≤ 0.06	0.08
		24	Pb16	Cu16	≤ 0.06	0.13
		96	Pb18	Cu18	0.09	0.18
	pH 6.5 Extraction Water	0	Extraction Water		≤ 0.06	
		6	Pb11, Control	Cu11, Control	1.32	≤ 0.06
		96	Pb11, Control	Cu11, Control	0.42	0.13
		6	Pb12	Cu12	0.14	0.13
		6	Pb13	Cu13	0.14	0.12
		96	Pb12	Cu12	0.40	0.44
		96	Pb13	Cu13	0.46	0.43
		6	Pb14	Cu14	0.09	0.09
		96	Pb14	Cu14	0.43	0.55
SS Nipples Removed, Liners Cleaned, Then Fill-and-Dump Experiment 4 (FD-04)	pH 6.5 Extraction Water	0	Extraction Water		Not Analyzed	Not Analyzed
		6	Pb11, Control	Cu11, Control		
		96	Pb11, Control	Cu11, Control		
		6	Pb19	Cu19		
		96	Pb19	Cu19		
		6	Pb12	Cu12		
		6	Pb13	Cu13		
		96	Pb12	Cu12		
		96	Pb13	Cu13		
		6	Pb14	Cu14		
		96	Pb14	Cu14		

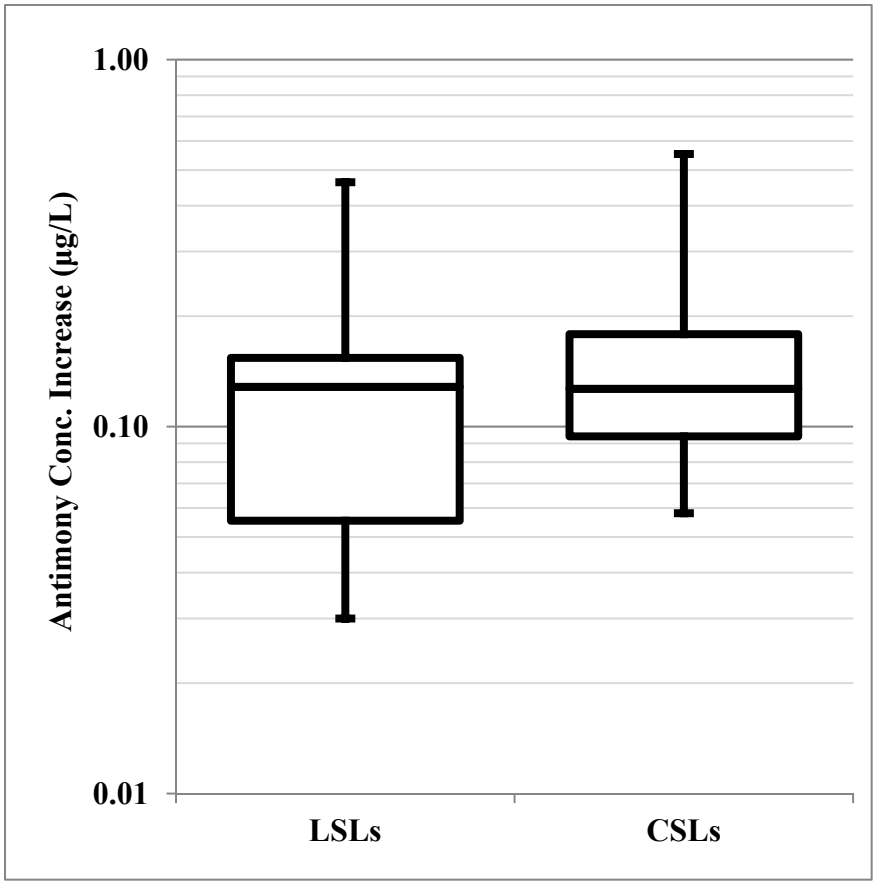

Figure 19: Antimony concentration increases in extraction water samples exposed to PET-lined LSL (n = 13) and CSL (n = 13) pipe specimens

Table 11: Mean antimony (Sb) levels in samples exposed to PET-lined pipe specimens

Extraction Water	Sample Subset	Mean Sb Concentration (µg/L)	±SD (µg/L)	Number of Samples
Dechlorinated Tap Water	Initial	0.19	N/A	1
	Exposed to Lined Pipe Specimens	0.28	0.04	8
Chlorinated pH 8 Extraction Water	Initial	≤ 0.06	N/A	1
	Exposed to Lined Pipe Specimens	0.09	0.06	6
pH 6.5 Extraction Water	Initial	≤ 0.06	N/A	1
	Exposed to Lined Pipe Specimens	0.29	0.18	12

A careful inspection of the antimony data shows that the samples were not contaminated with antimony by water that leaked behind the liners as the pipes were flushed. First, the median antimony increase in the samples exposed to PET-lined LSLs is approximately the same as the median increase in samples collected from PET-lined CSLs (0.13 µg/L for both). Had water passing behind or through the PET liner leached a significant amount of antimony from the pipe walls and then passed into the samples, the antimony concentrations in samples from the PET-lined LSLs would have been higher than those in samples from the PET-lined CSLs, since there was a significant amount of antimony present in the unlined and uncoated LSLs, but not in the unlined and uncoated CSLs. Furthermore, if 10 – 1,000 µL of water (the same volume needed to contaminate the samples with lead, as discussed above) containing the highest antimony concentration increase observed in an unlined control pipe (3.94 µg/L) had circumvented a PET liner and contaminated a sample collected from a PET-lined pipe specimen, the resulting antimony concentration increase would have been undetectable (≤ 0.06 µg/L). The unreasonable water volume needed to contaminate extraction water samples to the levels observed, coupled with the consistency of antimony increase in PET-lined LSLs and CSLs as well as the inconsistency in antimony increase between the unlined LSL and CSL controls, indicates that there is no relationship between the pipe material and antimony increase and that the increase in antimony is the result of leaching from PET liner and not leaching from the pipe wall.

Although the PET liners leached small amounts of antimony, the concentrations were significantly lower (one-tailed t-test, $\alpha = 0.05$) than in extraction waters exposed to the unlined (control) LSL. PET liners can thus significantly reduce antimony concentrations in drinking water by providing a barrier between antimony-containing deposits and drinking water. Although other trace constituents observed in pipe scales were not analyzed for in this study, the PET liner would be expected to provide the same leaching barrier for them as well.

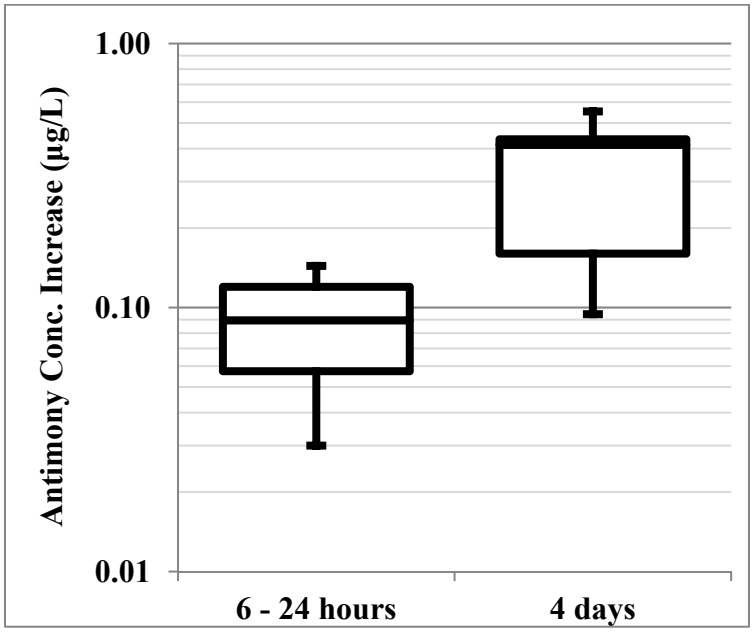

Figure 20: Antimony concentration increases in extraction water samples exposed to PET-lined pipe specimens for 6 – 24 hours (n = 16) and 4 days (n = 10)

Total Organic Carbon

Table 12 displays the TOC concentrations in samples collected from fill-and-dump experiments on PET-lined pipe specimens. As displayed in Figure 21 and Table 13, the average TOC concentrations in the dechlorinated tap water and chlorinated pH 8.0 extraction water were 3.34 mg/L (n = 1) and 0.15 mg/L (n = 1), respectively. The measured TOC concentration was below the detection limit in the pH 6.5 extraction water and in 10 of the 12 samples of pH 6.5 extraction water exposed to PET-lined pipes; and the two remaining samples contained only 0.11 mg/L of TOC. The mean TOC concentrations in dechlorinated tap water and chlorinated pH 8.0 extraction water samples exposed to PET-lined pipes were 3.37 ± 0.03 mg/L (n = 8) and 0.30 ± 0.08 mg/L (n = 6), respectively. There was no significant increase (one-tailed t-test, $\alpha = 0.05$) in TOC concentration in samples collected from PET-lined pipes exposed to dechlorinated tap water and pH 6.5 extraction water. A very slight mean TOC concentration increase of 0.15 ± 0.08 mg/L (n = 6) was observed in samples collected from PET-lined pipes exposed to chlorinated pH 8.0 extraction water. This increase was statistically significant (one-tailed test, $\alpha = 0.05$); however, since both the initial TOC concentration (0.15 mg/L) and the average concentration in the samples exposed to the PET-lined pipe specimens (0.30 mg/L) were so low, it is possible that the observed increase is associated with sample handling or small variations in instrument response rather than leaching.

The TOC data are deemed to be valid despite the fact water may have passed behind the liners as the lined specimens were flushed. TOC concentrations increased in extraction waters exposed to the unlined (control) specimens by a maximum of 1.09 mg/L. Unreasonably high volumes of contaminated water (from behind the liner) would have been needed to achieve the TOC increases observed in the chlorinated pH 8.0 extraction water. For example, it would have taken approximately 10 mL of contaminated water containing 1.09 mg/L TOC (the highest observed value in the unlined control) to contaminate 220 mL (the average volume of lined LSLs) of extraction water with 0.05 mg/L TOC, a smaller increase than observed in all but one of the chlorinated pH 8.0 extraction water samples. Furthermore, there was no detectable TOC increase observed in samples obtained using dechlorinated tap water and pH 6.5 extraction water, whereas all but one sample collected from each unlined control pipe (LSL and CSL) exhibited an increase in TOC concentration. These findings indicate that the samples collected from lined pipes were not affected by the water that may have passed behind the liners.

Table 12: TOC concentrations in samples collected from fill-and-dump experiments on PET-lined pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		TOC, mg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 3 (FD-03)	Dechlorinated Tap Water, pH 8	0	Extraction Water		3.34	
		6	Pb11, Control	Cu11, Control	3.34	3.21
		6	Pb12	Cu12	3.35	3.36
		6	Pb13	Cu13	3.36	3.34
		24	Pb15	Cu15	3.45	3.36
		96	Pb17	Cu17	3.37	3.38
	Chlorinated pH 8 Extraction Water	0	Extraction Water		0.15	
		6	Pb14	Cu14	0.22	0.19
		24	Pb16	Cu16	0.36	0.27
		96	Pb18	Cu18	0.39	0.35
	pH 6.5 Extraction Water	0	Extraction Water		≤ 0.10/≤ 0.10*	
		6	Pb11, Control	Cu11, Control	0.38	0.16
		96	Pb11, Control	Cu11, Control	1.09	0.18
		6	Pb12	Cu12	≤ 0.10	≤ 0.10
		6	Pb13	Cu13	0.11	≤ 0.10
		96	Pb12	Cu12	0.11	≤ 0.10
		96	Pb13	Cu13	≤ 0.10	≤ 0.10
		6	Pb14	Cu14	≤ 0.10	≤ 0.10
		96	Pb14	Cu14	≤ 0.10	≤ 0.10
Stainless Steel Nipples Removed, Liners Cleaned, Then Fill-and-Dump Experiment 4 (FD-04)	pH 6.5 Extraction Water	0	Extraction Water		Not Analyzed	Not Analyzed
		6	Pb11, Control	Cu11, Control		
		96	Pb11, Control	Cu11, Control		
		6	Pb19	Cu19		
		96	Pb19	Cu19		
		6	Pb12	Cu12		
		6	Pb13	Cu13		
		96	Pb12	Cu12		
		96	Pb13	Cu13		
		6	Pb14	Cu14		
		96	Pb14	Cu14		

* The FD-03 pH 6.5 extraction waters used in the 6 hour and 4 day experiments each contained ≤ 0.10 mg/L TOC

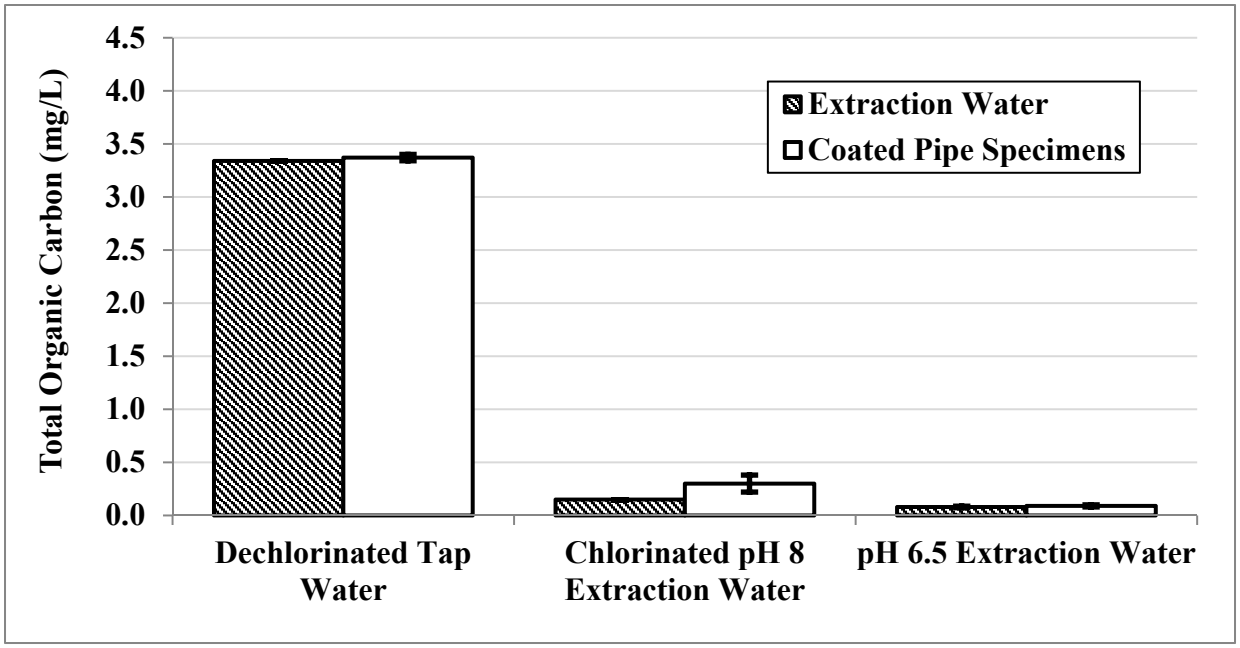

Figure 21: TOC concentrations in extraction waters and in samples collected from PET-lined pipe specimens (Error bars represent SD)

Table 13: Mean total organic carbon (TOC) levels in samples exposed to PET-lined pipes

Extraction Water	Sample Subset	Mean TOC Concentration (mg/L)	±SD (mg/L)	Number of Samples
Dechlorinated Tap Water	Initial	3.34	N/A	1
	Exposed to Lined-Pipe Specimen	3.37	0.03	8
Chlorinated pH 8 Extraction Water	Initial	0.15	N/A	1
	Exposed to Lined-Pipe Specimen	0.30	0.08	6
pH 6.5 Extraction Water	Initial	≤ 0.10	0.01	2
	Exposed to Lined-Pipe Specimen	≤ 0.10	0.02	12

Residual Chlorine

Substantially less free chlorine was consumed in PET-lined pipe specimens than in unlined control pipe specimens when exposed to chlorinated pH 8.0 extraction water, as displayed in Figure 12 (control specimens) and in Figure 22 (PET-lined specimens), the data for which can be found in Table 30 in Appendix E. In the PET-lined pipe specimens, approximately half of the initial free chlorine of 2 mg/L as Cl_2 was consumed in 96 hours.

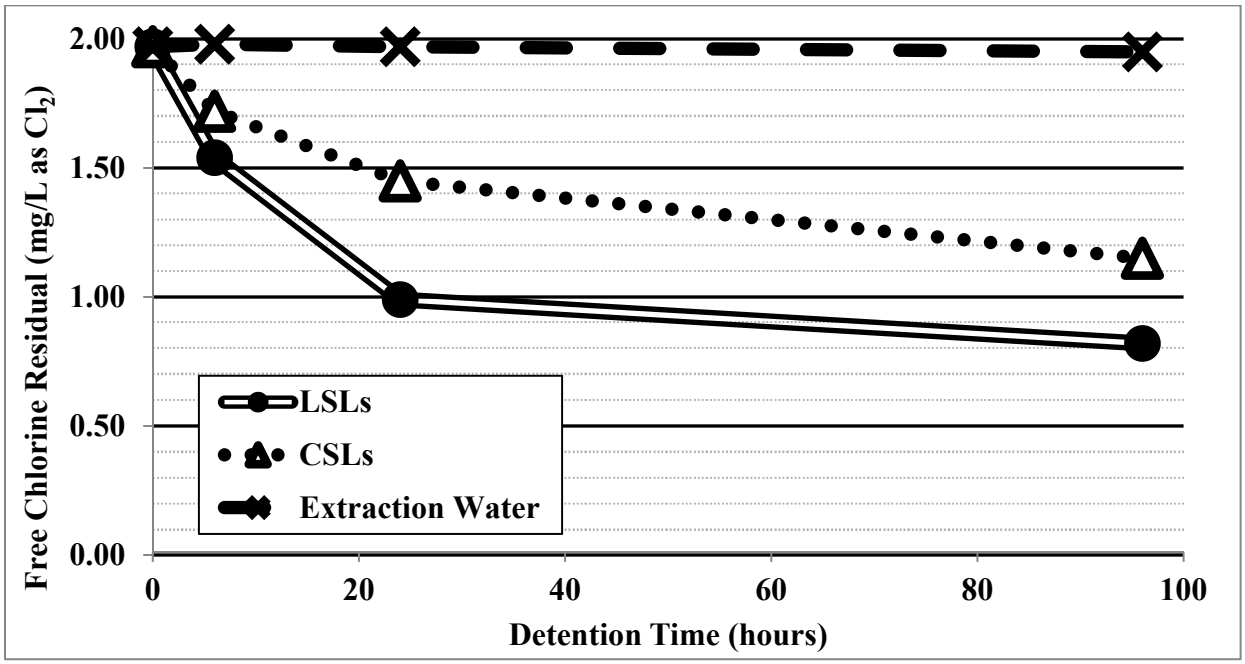

Figure 22: Free chlorine remaining over time in PET-lined pipe specimens exposed to chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of 1.97 mg/L free chlorine as Cl_2

Free chlorine demand diminished after repeated exposure to chlorinated extraction water, as seen in Figure 23. After three 24-hour fill-and-dump experiments with chlorinated pH 8.0 extraction water dosed with 2 mg/L free chlorine as Cl_2, approximately 0.1 mg/L Cl_2, or 5% of the initial free chlorine, was consumed in the PET-lined pipe specimens.

The PET liner material appears not to be reactive with free chlorine. When a sample piece of unexpanded PET liner (not installed) was exposed to chlorinated water for the first time, it consumed only 10% of the initial chlorine dose (2 mg/L as Cl_2) after 24 hours, as seen in Figure 23. (This sample was exposed only twice, during the second and third rounds of testing, not during the first round.) The PET-lined pipe specimens consumed more chlorine during their first exposure, but the demand quickly diminished to the levels of the unused PET liner after two or three more exposures as shown in Figure 23 (the data for which can be found in Table 31 in Appendix E). The initial chlorine demand exhibited by the PET-lined pipe specimens is likely due to minute amounts of impurities introduced during installation or flushing. These impurities, if present, were likely oxidized or flushed during the initial exposure, allowing subsequent tests to be more representative of the actual PET liner's chlorine demand.

Since the chlorine demand exerted by the PET liner is smaller than that for unlined control pipes, the potential for DBP formation should also be smaller. Thus, PET liners can potentially reduce both chlorine demand and DBP formation in the lead and copper service lines. Since the PET liner exhibited no significant long-term chlorine demand and no significant leaching of TOC, the data obtained in this study provide no reason to suspect that a PET liner would contribute to DBP formation in a service line.

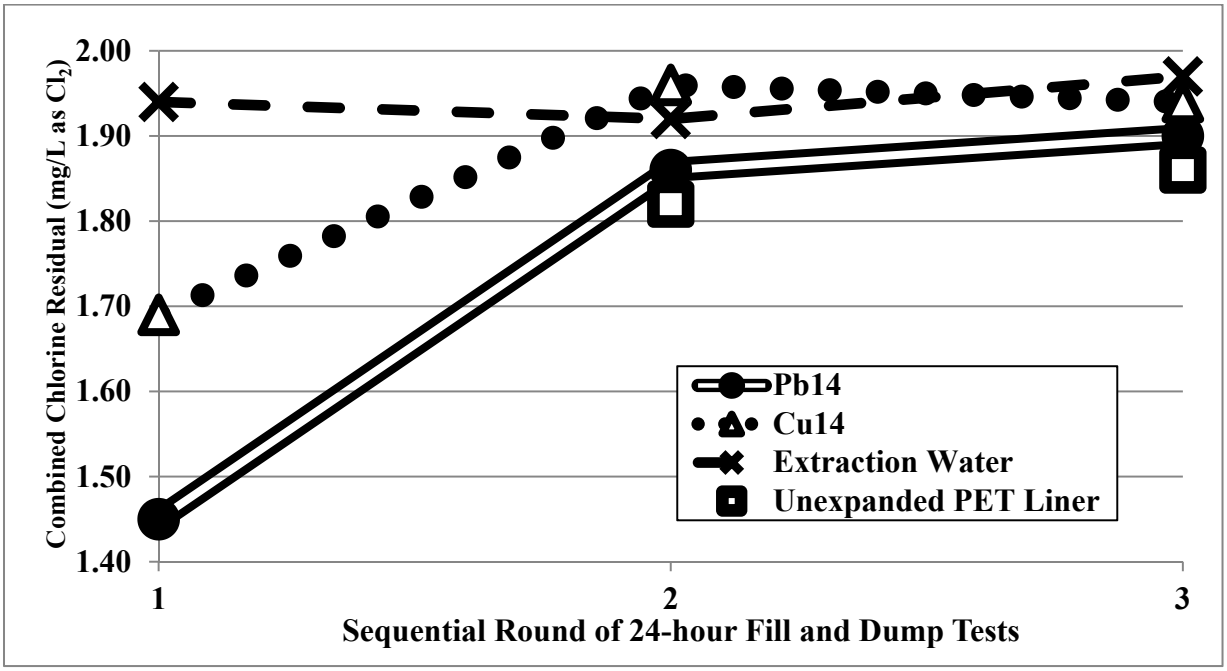

Figure 23: Free chlorine demand of PET-lined pipe specimens and extraction water after sequential 24-hour fill-and-dump experiments with chlorinated pH 8.0 extraction water dosed with sodium hypochlorite to an initial concentration of approximately 2.00 mg/L as Cl_2

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

Lead levels in epoxy-coated lead pipes were dramatically lower (3 orders of magnitude) than those in the uncoated lead control pipe. Only one sample (out of 27 total) collected from epoxy-coated LSLs contained a lead concentration (78 μg/L) above the AL of 15 μg/L, and subsequent samples from the same pipe contained no detectable lead (≤ 0.5 μg/L), as did the majority of samples (16 out of 27). Of the 11 samples containing detectable lead, 7 were collected from the same pipe specimen, Pb05, which may have been compromised by damage to the end-fittings, since detectable levels of lead were consistently found in samples from this pipe specimen. Even so, lead levels in this pipe specimen were well below the action level.

An elemental analysis using ICP-MS and GFAAS on partially digested epoxy revealed that it contained approximately 8 mg copper/kg epoxy. The copper content may have contributed to consistently detectable levels of copper (≥ 0.25 μg/L) in extraction waters exposed to epoxy-coated pipes. However, copper levels in extraction waters exposed to epoxy-coated pipes were dramatically less (1 – 2 orders of magnitude) than those in the uncoated copper control pipe. Although 48 of 51 samples collected from epoxy-coated pipes (both LSLs and CSLs) exhibited a detectable increase in copper concentration (0.4 – 22 μg/L) compared to initial extraction water, the highest observed concentrations were negligible relative to the AL for copper (1,300 μg/L). There appears to be no reason for utilities or consumers to be concerned about the copper present in the epoxy.

The epoxy-coated pipes also exhibited a substantial demand for both free and combined chlorine and leached a small but significant ($\alpha = 0.05$) amount of TOC into chlorinated pH 8 and pH 6.5 extraction waters (0.65 mg/L ± 0.62, n = 44) but not into dechlorinated tap water. After repeated exposure to free chlorine, the 1-hour free chlorine demand persisted, but gradually decreased to about 10% of the initial chlorine concentration. With epoxy coatings being reactive with free and combined chlorine, research is recommended to investigate the long-term structural and leaching effects that may occur. Furthermore, since TOC leaching and chlorine demand have been observed, the potential for DBP formation in extraction waters exposed to an epoxy coating needs to be investigated.

An elemental analysis using ICP-MS on digested PET liner material indicated it contained one element of concern, antimony (MCL = 6 μg/L). The average antimony concentration in three samples of digested PET liner was approximately 130 mg Sb/kg PET. The presence of antimony in the PET liner prompted monitoring in extraction waters exposed to PET-lined pipe specimens. Antimony concentrations in samples collected from the PET-lined pipes increased by an average of only 0.18 ± 0.16 μg/L (n = 26) and consistently remained more than an order of magnitude below the MCL for antimony. Antimony in samples exposed to PET-lined pipes 6 – 24 hours was significantly greater (one-tailed t-test, $\alpha = 0.05$) than initial levels in their corresponding extraction waters, for a mean increase of 0.09 ± 0.04 μg/L (n = 16). Antimony concentrations in samples of extraction water exposed to PET-lined pipes for 4 days were significantly greater (one-tailed t-test, $\alpha = 0.05$) than those in samples exposed 6 – 24 hours, with a mean increase of 0.33 ± 0.17 μg/L (n = 10) over initial levels. Samples collected from the unlined (control) LSL contained higher amounts of antimony (0.42 – 3.94 μg/L),

leached from deposits in the LSL, than samples from the PET-lined LSLs. Therefore, despite leaching minute quantities of antimony into the extraction water, a PET liner can potentially reduce antimony levels in tap water by preventing antimony from leaching out of deposits.

The PET-lined pipes exhibited an initial free chlorine demand of only about 1 mg/L over 4 days with an initial free chlorine concentration of 2 mg/L as Cl_2. When the same PET-lined pipes were again exposed to free chlorine for two or three 24-hour periods, the 24-hour chlorine demand decreased to about 5 percent of the initial chlorine concentration, approximately the same as the 24-hour demand found in extraction water stored in the dark in an amber glass, stoppered reservoir and used as a control. Thus the PET liner exhibited no significant long-term chlorine demand.

A very small (0.15 ± 0.08 mg/L $n = 6$) but statistically significant (one-tailed t-test, $\alpha = 0.05$) increase in TOC concentration was observed in one of three extraction waters (chlorinated pH 8 extraction water) exposed to PET-lined pipe specimens. However, the average TOC concentration in the samples of exposed pH 8 extraction water was only 0.30 mg/L, and at a TOC concentration this low, the small increase observed may have been associated with sample handling or small variations in instrument response rather than leaching. No increase was observed using the other extraction waters, including dechlorinated tap water. Since the PET liner exhibited no significant long-term chlorine demand and no significant leaching of TOC, there is no reason to suspect that a PET liner would contribute to DBP formation in a service line.

There were only two products tested in this study, an epoxy coating and a PET liner. Both products are commercially available and certified for use in water service lines, but there are other lining and coating materials currently in use, or being considered for use, in service lines, such as other epoxy formulations, polyurethanes, and polyureas. More research is needed on these other materials (and some research has already been done or is in progress, under the auspices of the manufacturers themselves) to examine their effectiveness in preventing the release of lead and copper, over both short and long periods of time, and their effects on drinking water quality, specifically the organic and inorganic chemicals that may leach into the drinking water. Fill-and-dump experiments can provide a reproducible method to study the worst-case scenarios experienced by service lines that are either lined or coated. By stagnating the extraction water, chemicals of concern are allowed to accumulate, simulating potential household scenarios. If contaminants are not detected or are detected only at levels far below the levels of concern, this helps reassure both utilities and consumers; and if higher levels of contaminants are found, the results can be used as the basis for a preliminary risk assessment or to guide further experiments to measure exposure under more typical conditions.

APPENDIX A – LEAD AND COPPER RULE REFERENCE GUIDE

Lead and Copper Rule: A Quick Reference Guide

Overview of the Rule	
Title[1]	Lead and Copper Rule (LCR)[2], 56 FR 26460 - 26564, June 7, 1991
Purpose	Protect public health by minimizing lead (Pb) and copper (Cu) levels in drinking water, primarily by reducing water corrosivity. Pb and Cu enter drinking water mainly from corrosion of Pb and Cu containing plumbing materials.
General Description	Establishes action level (AL) of 0.015 mg/L for Pb and 1.3 mg/L for Cu based on 90th percentile level of tap water samples. An AL exceedance is not a violation but can trigger other requirements that include water quality parameter (WQP) monitoring, corrosion control treatment (CCT), source water monitoring/treatment, public education, and lead service line replacement (LSLR).
Utilities Covered	All community water systems (CWSs) and non-transient non-community water systems (NTNCWSs) are subject to the LCR requirements.

Public Health Benefits	
Implementation of the LCR has resulted in	▶ Reduction in risk of exposure to Pb that can cause damage to brain, red blood cells, and kidneys, especially for young children and pregnant women. ▶ Reduction in risk of exposure to Cu that can cause stomach and intestinal distress, liver or kidney damage, and complications of Wilson's disease in genetically predisposed people.

Lead Consumer Notice
Within 30 days of learning the results, all systems must provide individual Pb tap results to people who receive water from sites that were sampled, *regardless of whether the results exceed the Pb AL*, as required by 40 CFR 141.85(d).

Consumer Confidence Report (CCR)
All CWSs, irrespective of their lead levels, must provide an educational statement about lead in drinking water in their CCRs as required by 40 CFR 141.154. Must be in 2008 CCR (due July 1, 2009) if EPA is Primacy Agency, State adopts the rule by reference automatically, or adopts during 2008. Otherwise, this statement is required in the 2009 CCR (due July 1, 2010).

[1]This document provides a summary of federal drinking water requirements; to ensure full compliance, please consult the federal regulations at 40 CFR 141 and any approved state requirements.

[2] The June 1991 LCR was revised with the following Technical Amendments: 56 FR 32112, July 15, 1991; 57 FR 28785, June 29, 1992; 59 FR 33860, June 30, 1994.

It was subsequently revised by: the LCR Minor Revisions, 65 FR 1950, January 12, 2000; and the LCR Short-Term Revisions, 72 FR 57782, October 10, 2007.

APPENDIX A – LEAD AND COPPER RULE REFERENCE GUIDE

Major Monitoring Provisions

Lead and Copper Tap

Applicability	▶ All CWSs and NTNCWSs.
Standard	▶ CWSs and NTNCWSs must collect first-draw samples at taps in homes/buildings that are at high risk of Pb/Cu contamination as identified in 40 CFR 141.86(a). ▶ Number of samples is based on system size (see Table 1). ▶ Systems must conduct monitoring every 6 months unless they qualify for reduced monitoring.
Reduced	▶ See Table 1 for sample number and Table 2 for criteria.

Water Quality Parameter (WQP)

Applicability	▶ Systems serving > 50,000 people. ▶ Systems serving ≤ 50,000 during monitoring periods in which either AL is exceeded.
Standard	▶ WQP samples at taps are collected every 6 months. ▶ WQPs at entry points to distribution system (EPTDS) are collected every 6 months prior to CCT installation, then every 2 weeks.
Reduced	▶ See Table 1 for sample number and page 2 for criteria. Does not apply to EPTDS WQP monitoring.

Table 1: Lead and Copper Tap and WQP Tap Monitoring

Size Category	System Size	Number of Pb/Cu Tap Sample Sites[3]		Number of WQP Tap Sample Sites[4]	
		Standard	Reduced	Standard	Reduced
Large	> 100K	100	50	25	10
	50,001 - 100K	60	30	10	7
Medium	10,001 - 50K	60	30	10	7
	3,301 - 10K	40	20	3	3
Small	501 - 3,300	20	10	2	2
	101 - 500	10	5	1	1
	≤ 100	5	5	1	1

[3] With written State approval, PWSs can collect < 5 samples if all taps used for human consumption are sampled.
[4] Two WQP tap samples are collected at each sampling site.

Table 2: Criteria for Reduced Pb/Cu Tap Monitoring

Annual	1. PWS serves ≤ 50,000 people and is ≤ both ALs for 2 consecutive 6-month monitoring periods; or 2. Any PWS that meets optimal WQPs (OWQPs) and is ≤ Pb AL for 2 consecutive 6-month monitoring periods.
Triennial	1. PWS serves ≤ 50,000 people and is ≤ both ALs for 3 consecutive years of monitoring; or 2. Any PWS that meets OWQP specifications and is ≤ Pb AL for 3 consecutive years of monitoring; or 3. Any PWS with 90th percentile Pb and Cu levels ≤ 0.005 mg/L and ≤ 0.65 mg/L, respectively, for 2 consecutive 6-month monitoring periods (i.e., accelerated reduced Pb/Cu tap monitoring).
Every 9 years	PWS serves ≤ 3,300 people and meets monitoring waiver criteria found at 40 CFR 141.86(g).

APPENDIX A – LEAD AND COPPER RULE REFERENCE GUIDE

Treatment Technique and Sampling Requirements if the AL is Exceeded[5]	
[5] Based on 90th percentile level. Multiply number of valid samples by 0.9 (e.g., 10 samples x 0.9 = 9; thus, use 9th highest Pb and Cu test result to compare to AL). For 5 samples, average 4th and 5th highest results. For < 5 samples, use highest result.	
Water Quality Parameter (WQP)	
Applicability	Refer to page 1.
Parameters	► pH, alkalinity, calcium *(initial only, unless calcium carbonate stabilization is used)*, conductivity *(initial monitoring only)*, orthophosphate *(if inhibitor is phosphate-based)*; silica *(if inhibitor is silicate-based)*, and temperature *(initial monitoring only)*.
Frequency	► Systems installing CCT, must conduct follow-up monitoring for 2 consecutive 6-month periods. ► WQP tap monitoring is conducted every 6 months, EPTDS monitoring increases to every 2 weeks. ► After follow-up monitoring, State sets OWQP specifications that define optimal CCT.
Reduced Tap Monitoring	► Collect reduced number of sampling sites (see Table 1) if meet OWQPs for 2 consecutive 6-month periods. ► Collect reduced number of sampling sites at reduced frequency if meet OWQPs for: – 6 consecutive 6-month monitoring periods can monitor annually; – 3 consecutive years of annual monitoring can monitor triennially.
Public Education (PE)	
Applicability	► Systems that exceed the Pb AL (*not required if only the Cu AL is exceeded*).
Purpose	► Educates consumers about lead health effects, sources, and steps to minimize exposure.
Delivery Method	► CWSs: deliver materials to bill-paying customers and post lead information on water bills, work in concert with local health agencies to reach at-risk populations (children, pregnant woman), deliver to other organizations serving "at-risk" populations, provide press releases, include new outreach activities from list in 40 CFR 141.85(a)(2)(vi), and post to Web site (CWSs serving > 100,000 only). ► NTNCWSs: posting and distribution to all consumers (can be electronic with State permission). Can apply to CWSs such as hospitals and prisons where population cannot make improvements.
Timing	► Within 60 days *after end of monitoring period* in which Pb AL was exceeded if not already delivering PE.[6] ► Repeat annually except: water bill inserts - quarterly; press releases - 2x/year, and Web posting - continuous. ► Can discontinue whenever ≤ Pb AL but must recommence if subsequently exceed Pb AL.
[6] State may allow extension in some situations. Also, State may require approval of message content prior to delivery.	

APPENDIX A – LEAD AND COPPER RULE REFERENCE GUIDE

Treatment Technique and Sampling Requirements if the AL is Exceeded[5]	
[5] Based on 90th percentile level. Multiply number of valid samples by 0.9 (e.g., 10 samples x 0.9 = 9; thus, use 9th highest Pb and Cu test result to compare to AL). For 5 samples, average 4th and 5th highest results. For < 5 samples, use highest result.	
Source Water Monitoring and Source Water Treatment (SOWT)	
Applicability	▶ Systems that exceed Pb or Cu AL.
Purpose	▶ Determine contribution from source water to total tap water Pb and Cu levels and need for SOWT.
Timing	▶ One set of samples at each EPTDS is due within 6 months of first AL exceedance. ▶ System has 24 months to install any required SOWT. ▶ State sets maximum permissible levels (MPLs) for Pb and Cu in source water based on initial and follow-up source water monitoring.
Standard	▶ Ground water PWSs monitor once during 3-year compliance periods; surface water PWSs monitor annually.
Reduced	▶ Monitor every 9 years if MPLs are not exceeded during 3 consecutive compliance periods for ground water PWSs or 3 consecutive years for surface water PWSs.
Corrosion Control Treatment (CCT)	
Applicability	▶ All large systems except those meeting requirements of 40 CFR 141.81(b)(2) or (b)(3). ▶ Medium and small systems that exceed either AL; may stop CCT steps if ≤ both ALs for 2 consecutive 6-month periods but must recommence CCT if subsequently exceed either AL.
Study	▶ All large systems except as noted above. ▶ If State requires study for small or medium systems, it must be completed within 18 months.
Treatment	▶ Once State determines type of CCT to be installed, PWS has 24 months to install. ▶ Systems installing CCT must conduct 2 consecutive 6 months of follow-up tap and WQP monitoring.
OWQPs	▶ After follow-up Pb/Cu tap and WQP monitoring, State sets OWQPs. *Refer to WQP section above.*
Lead Service Line Replacement (LSLR)	
Applicability	▶ Systems that continue to exceed the Pb AL after installing CCT and/or SOWT. ▶ Can discontinue LSLR whenever ≤ Pb AL in tap samples for 2 consecutive 6-month monitoring periods; must recommence if subsequently exceed.
Monitoring	▶ Optional: Sample from LSL to determine if line must be replaced. If all samples are ≤ 0.015 mg/L, line is considered "replaced through testing"; must reconsider these lines if Pb AL is subsequently exceeded. ▶ Required: Sample from any LSLs not completely replaced to determine impact on Pb levels.
Replacement	▶ Must replace at least 7% of LSLs annually; State can require accelerated schedule. ▶ If only portion of LSL is replaced, PWS must: – Notify customers at least 45 days prior to replacement about potential for increased Pb levels. – Collect samples within 72 hours of replacement and provide results within 3 days of receipt.

EPA 816-F-08-018 http://water.epa.gov/drink June 2008

(EPA 2008)

APPENDIX B – CITY OF LAWRENCE, KAN. DRINKING WATER CONSUMER CONFIDENCE REPORT

Testing Results for: City of Lawrence

(During the 2011 calendar year, we had no violations of drinking water regulations)

Microbiological	Result	MCL	MCLG	Typical Source
A minimum of 90 samples are collected and tested each month throughout the distribution system. No Detected Results were Found in the Calendar Year of 2011.				

Regulated Contaminants	Collection Date	Your Highest Value	Range (low/high)	Unit	MCL	MCLG	Typical Source
ASBESTOS	8/19/2004	ND	NA	MF/L	7	7	Decay of asbestos water main
ARSENIC	8/17/2011	3.8	ND - 3.8	ppb	10	0	Erosion of natural deposits
ATRAZINE	6/1/2011	0.3	ND - 0.3	ppb	3	3	Runoff from herbicide used on row crops
BARIUM[1]	2/16/2011	0.120	0.019 - 0.120	ppm	2	2	Discharge from metal refineries
FLUORIDE	8/17/2011	0.97	0.2 - 0.97	ppm	4	4	Erosion of natural deposits; water additive which promotes strong teeth
NITRATE	2/16/2011	1.3	ND - 1.3	ppm	10	10	Runoff from fertilizer use
SELENIUM	2/16/2011	3.4	ND - 3.4	ppb	50	50	Erosion of natural deposits
TURBIDITY[2]	4/22/2011	0.395	0.010- 0.395	NTU	1		Soil runoff
CHLORAMINE	2011	3.6 (RAA)	3.5 – 3.6	ppm	4 (MRDL)	4 (MRDLG)	Additive to control microbes
TOTAL ORGANIC CARBON[3]	2011	3.75 (RAA)	2.93 -3.75	ppm	TT	NA	Naturally present in the environment

1. Some people who drink water containing barium in excess of the MCL over many years could experience an increase in their blood pressure.
2. Turbidity has no health effects. However, turbidity can interfere with disinfection and provide a medium for microbial growth. Turbidity may indicate the presence of disease-causing organisms. These organisms include bacteria, viruses, and parasites that can cause symptoms such as nausea, cramps, diarrhea and associated headaches.
3. Total organic carbon has no health effects. However, total organic carbon provides a medium for the formation of disinfection byproducts. These byproducts include Total Trihalomethanes and Total Haloacetic acids.

Disinfection Byproducts	Monitoring Period	Your Highest RAA	Range (low/high)	Unit	MCL	MCLG	Typical Source
TOTAL HALOACETIC ACIDS	2011	29.8	15.4 - 32.7	ppb	60	0	By-product of drinking water disinfection
TOTAL TRIHALOMETHANES	2011	51.8	23.1 - 63.5	ppb	80	0	By-product of drinking water chlorination

Drinking water containing these byproducts in excess of the MCL may lead to adverse health effects, liver or kidney problems, or nervous system effects, and may lead to an increased risk of getting cancer.

Lead and Copper	Monitoring Period	90th Percentile	Range (low/high)	Unit	AL	Sites Over AL	Typical Source
COPPER, FREE	2011 - 2013	0.086	0.0063 - 0.12	ppm	1.3	0	Corrosion of household plumbing
LEAD	2011 - 2013	2.7	ND - 9.9	ppb	15	0	Corrosion of household plumbing

If present, elevated levels of lead can cause serious health problems, especially for pregnant women and young children. Lead in drinking water is primarily from materials and components associated with service lines and home plumbing. Your water system is responsible for providing high quality drinking water, but cannot control the variety of materials used in plumbing components. When your water has been sitting for several hours, you can minimize the potential for lead exposure by flushing your tap for 30 seconds to 2 minutes before using water for drinking or cooking. If you are concerned about lead in your water, you may wish to have your water tested. Information on lead in drinking water, testing methods, and steps you can take to minimize exposure is available from the Safe Drinking Water Hotline or at http://www.epa.gov/safewater/lead.

APPENDIX B – CITY OF LAWRENCE, KAN. DRINKING WATER CONSUMER CONFIDENCE REPORT

Testing Results for: City of Lawrence

(During the 2011 calendar year, we had no violations of drinking water regulations)

Radiological Contaminants	Collection Date	Your Highest Value	Range (low/high)	Unit	MCL	MCLG	Typical Source
COMBINED RADIUM (226 & 228)	11/10/2008	1	ND - 1	pCi/L	5	0	Erosion of natural deposits
GROSS ALPHA	11/10/2008	3	3	pCi/L	15	0	Erosion of natural deposits

Constituents Having Secondary MCL's	Collection Date	Your Highest Value	Range (low/high)	Unit	SMCL
ALUMINUM	5/11/2011	0.23	0.012 - 0.23	ppm	0.05
CHLORIDE	2/16/2011	100	13 - 100	ppm	250
CORROSIVITY	5/11/2011	0.22	0.19 - 0.22	Langelier Index	0
Hydrogen ion (pH)	11/16/2011	9.9	8.0 - 9.9	S.U.	8.5
SULFATE	2/16/2011	150	27 - 150	ppm	250
Total Dissolved Solids (TDS)	2/16/2011	500	160 - 500	ppm	500

UNREGULATED PARAMETERS	FEDERAL LEVEL RECOMMENDED	Your Highest Value	Range (low/high)	Unit	TYPICAL SOURCE
Unregulated Parameters are monitored in the interest of the customers and to assist regulators in developing future regulations					
ALKALINITY, TOTAL as $CaCO_3$	300	120	40 - 120	ppm	Erosion of natural deposits
CALCIUM	200	43	24 - 43	ppm	Erosion of natural deposits
CONDUCTIVITY @ 25 °C	1500	1300	310 - 1300	µmhos/cm	Erosion of natural deposits
HARDNESS, TOTAL (AS CaC03)	400	190	100 - 190	ppm	Erosion of natural deposits
MAGNESIUM	150	20	9.3 - 20	ppm	Erosion of natural deposits
N-nitrosodimethylamine	NA	0.0024	ND – 0.0024	ppb	Byproduct of manufacturing of leather, rubber, and plastics. Reactions of precursor amines with nitrosating agents
NICKEL	0.1	0.0019	ND - 0.0019	ppm	Erosion of natural deposits
ORTHOPHOSPHATE	NA	0.2	ND - 0.2	ppm	Additive to control pipe corrosion
PHOSPHORUS, TOTAL	5	0.36	ND - 0.36	ppm	Erosion of natural deposits
POTASSIUM	100	11	2.9 - 11	ppm	Erosion of natural deposits
RADON	NA	14	ND - 14	pCi/L	Erosion of natural deposits
SILICA	50	15	1.3 - 15	ppm	Erosion of natural deposits
SODIUM	100	100	15 - 100	ppm	Erosion of natural deposits

(City of Lawrence 2012)

APPENDIX C – EXPERIMENTAL ARTIFACTS FROM END-FITTING MATERIALS

Table 14: Lead concentrations in lead solution and reagent water samples exposed to end-fitting materials for 7 d

Material	Replicate No.	Lead Concentration, µg/L			
		Lead Solution		Reagent Water	
		Initial	After Exposure to Material	Initial	After Exposure to Material
HTFE Tape	1	18.1	18.4	≤ 0.5	≤ 0.5
	2		18.5		
Threaded SS Nipple	1	19.5	19.4	≤ 0.5	≤ 0.5
	2		19.9		
Unthreaded SS Nipple	1	19.5	19.2	≤ 0.5	≤ 0.5
	2		19.3		
Silicone Stopper	1	14.2	14.0	Not Analyzed*	
	2	12.7	13.9		
HDPE Stopper	1	17.7	17.9	≤ 0.5	0.6
	2		18.4		

*Since no lead was detected in an ICP-MS analysis of a silicone stopper, only adsorption was tested and not leaching.

Table 15: Copper concentrations in copper solution and reagent water samples exposed to end-fitting materials for 7 d

Material	Replicate No.	Copper Concentration, µg/L			
		Copper Solution		Reagent Water	
		Initial	After Exposure to Material	Initial	After Exposure to Material
HTFE Tape	1	6.34	6.69	≤ 0.25	≤ 0.25
	2		6.62		
Threaded SS Nipple	1	5.98	19.7	≤ 0.25	2.28
	2		32.1		
Unthreaded SS Nipple	1	5.98	19.6	≤ 0.25	8.28
	2		21.7		
Silicone Stopper	1	24.9	25.5	Not Analyzed*	
	2	25.5	24.6		
HDPE Stopper	1	4.96	5.26	≤ 0.25	≤ 0.25
	2		5.10		

*Since no copper was detected in an ICP-MS analysis of a silicone stopper, only adsorption was tested and not leaching.

APPENDIX C – EXPERIMENTAL ARTIFACTS FROM END-FITTING MATERIALS

Table 16: Antimony concentrations in antimony solution and reagent water samples exposed to end-fitting materials for 7 d

		Antimony Concentration (μg/L)	
Material	Replicate No.	Antimony Solution*	Reagent Water**
HDPE Stopper	1	11.15	≤ 0.06
HDPE Stopper	2	11.10	
PTFE Tape	1	10.26	≤ 0.06
PTFE Tape	2	10.34	
PTFE Tape	3	10.78	
Unthreaded SS Nipple	1	7.59	0.09
Threaded SS Nipple	1	9.19	≤ 0.06

*Initial solution contained 10.23 μg/L antimony
**Reagent water initially contained ≤ 0.06 μg/L antimony

Table 17: Chlorine residuals in chlorinated pH 8 extraction water samples exposed to end-fitting materials for 24 h

Sample/Material	Replicate No.	Chlorine Residual (mg/L as Cl_2)
Extraction Water*	1	1.94
Silicone Stopper	1	1.91
	2	1.90
HDPE Stopper	1	1.96
	2	1.88
Glass Beaker**	1	1.94
	2	1.97

*Initial extraction water chlorine residual was 2.03 mg/L as Cl_2
**All materials tested were exposed to extraction water in 600 mL glass beakers. Glass beakers containing extraction water and no material were used as controls.

APPENDIX C – EXPERIMENTAL ARTIFACTS FROM END-FITTING MATERIALS

Table 18: TOC concentration in chlorinated pH 8 extraction water samples exposed to stopper materials for 24 h.

Material*	Replicate No.	TOC Concentration (mg/L)**
Glass Beaker Control	1	0.231
Glass Beaker Control	2	0.192
Silicone Stopper	1	0.185
Silicone Stopper	2	0.181
HDPE Stopper	1	0.160
HDPE Stopper	2	0.155
Reagent Blank	1	0.150
Reagent Blank	2	0.111

*All materials tested were exposed to extraction water in 600 mL glass beakers. Glass beakers containing extraction water and no material were used as controls.

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APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 19: Fill-and-dump testing matrix using epoxy-coated pipe specimens, listed in order of test

	Extraction Water	Holding Time, h	Pipe No.		Notes
			LSLs	CSLs	
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8	6	Pb04	Cu10	Uncoated Controls
		6	Pb01	Cu01	
		6	Pb02	Cu02	
		24	Pb05	Cu05	
		96	Pb07	Cu07	
		240	Pb05	Cu05	
	Chlorinated pH 8 Extraction Water	6	Pb03	Cu03	
		24	Pb06	Cu06	
		96	Pb08	Cu08	
		240	Pb06	Cu06	
	pH 6.5 Extraction Water	6	Pb04	Cu10	Uncoated Controls
		168	Pb04	Cu10	Uncoated Controls
		6	Pb01	Cu01	
		6	Pb02	Cu02	
		168	Pb01	Cu01	
		168	Pb02	Cu02	
		6	Pb03	Cu03	
		168	Pb03	Cu03	
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water	6	Pb04	Cu10	Uncoated Controls, Stored Dry
		168	Pb04	Cu10	Uncoated Controls
		6	Pb08	Cu08	Stored Dry
		24	Pb02	Cu02	Stored Wet
		24	Pb05	Cu05	Stored Dry
		24	Pb09	Cu09	Previously Unused
		168	Pb02	Cu02	
		168	Pb05	Cu05	
		168	Pb09	Cu09	
	Re-Flushed, then Chlorinated pH 8 Extraction Water	6	Pb02	Cu02	
		6	Pb05	Cu05	
		6	Pb08	Cu08	
		6	Pb09	Cu09	
		24	Pb02	Cu02	
		24	Pb05	Cu05	
		24	Pb08	Cu08	
		24	Pb09	Cu09	
		168	Pb02	Cu02	
		168	Pb05	Cu05	
		168	Pb08	Cu08	
		168	Pb09	Cu09	

APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 20: Lead concentration increases in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Pb, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1150	0.8
		6	Pb01	Cu01	78.3	≤ 0.5
		6	Pb02	Cu02	1.2	≤ 0.5
		24	Pb05	Cu05	3.8	0.6
		96	Pb07	Cu07	0.9	0.5
		240	Pb05	Cu05	0.8	0.7
	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb03	Cu03	≤ 0.5	≤ 0.5
		24	Pb06	Cu06	≤ 0.5	0.6
		96	Pb08	Cu08	1.2	≤ 0.5
		240	Pb06	Cu06	≤ 0.5	≤ 0.5
	pH 6.5 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1800	0.6
		168	Pb04, Control	Cu10, Control	25000	3.0
		6	Pb01	Cu01	≤ 0.5	≤ 0.5
		6	Pb02	Cu02	≤ 0.5	≤ 0.5
		168	Pb01	Cu01	≤ 0.5	≤ 0.5
		168	Pb02	Cu02	≤ 0.5	≤ 0.5
		6	Pb03	Cu03	≤ 0.5	≤ 0.5
		168	Pb03	Cu03	0.5	≤ 0.5
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb04, Control	Cu10, Control	1700	≤ 0.5
		168	Pb04, Control	Cu10, Control	2400	≤ 0.5
		6	Pb08	Cu08	≤ 0.5	≤ 0.5
		24	Pb02	Cu02	≤ 0.5	≤ 0.5
		24	Pb05	Cu05	2.11	≤ 0.5
		24	Pb09	Cu09	≤ 0.5	≤ 0.5
		168	Pb02	Cu02	≤ 0.5	≤ 0.5
		168	Pb05	Cu05	1.38	≤ 0.5
		168	Pb09	Cu09	≤ 0.5	≤ 0.5
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.5	
		6	Pb02	Cu02		
		6	Pb05	Cu05	6.2	
		6	Pb08	Cu08		
		6	Pb09	Cu09	≤ 0.5	≤ 0.5
		24	Pb02	Cu02		
		24	Pb05	Cu05	2.0	
		24	Pb08	Cu08		
		24	Pb09	Cu09	≤ 0.5	≤ 0.5
		168	Pb02	Cu02		
		168	Pb05	Cu05	1.3	
		168	Pb08	Cu08		
		168	Pb09	Cu09		

APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 21: Copper concentration increases in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Cu, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		5.7	
		6	Pb04, Control	Cu10, Control	8.3	380
		6	Pb01	Cu01	9.3	9.3
		6	Pb02	Cu02	3.4	14.3
		24	Pb05	Cu05	3.6	1.9
		96	Pb07	Cu07	-0.70	1.6
		240	Pb05	Cu05	-0.20	-2.6
	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb03	Cu03	6.8	7
		24	Pb06	Cu06	5.6	8.9
		96	Pb08	Cu08	10	15
		240	Pb06	Cu06	2.8	7.4
	pH 6.5 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb04, Control	Cu10, Control	5.5	830
		168	Pb04, Control	Cu10, Control	10	400
		6	Pb01	Cu01	2.6	6.3
		6	Pb02	Cu02	4.0	4.3
		168	Pb01	Cu01	7.1	15
		168	Pb02	Cu02	10	8.9
		6	Pb03	Cu03	2.1	1.9
		168	Pb03	Cu03	6.3	11
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb04, Control	Cu10, Control	3.7	270
		168	Pb04, Control	Cu10, Control	4.5	85
		6	Pb08	Cu08	2.7	3.3
		24	Pb02	Cu02	0.37	0.38
		24	Pb05	Cu05	2.6	1.3
		24	Pb09	Cu09	22	14
		168	Pb02	Cu02	1.1	1.0
		168	Pb05	Cu05	2.8	0.67
		168	Pb09	Cu09	4.1	3.8
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		≤ 0.25	
		6	Pb02	Cu02		
		6	Pb05	Cu05	2.2	
		6	Pb08	Cu08		
		6	Pb09	Cu09	2.8	8.7
		24	Pb02	Cu02		
		24	Pb05	Cu05	1.6	
		24	Pb08	Cu08		
		24	Pb09	Cu09	1.8	2.7
		168	Pb02	Cu02		
		168	Pb05	Cu05	2.0	
		168	Pb08	Cu08		
		168	Pb09	Cu09		

APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 22: Free chlorine residual in samples collected from uncoated control pipe specimens exposed to chlorinated pH 8 extraction water

Pipe No.		Holding Time, h	Free Chlorine Residual (mg/L as Cl_2)	
LSLs	CSLs		LSLs	CSLs
Pb04	Cu10	24	0.05	0.20
Pb04	Cu10	168	≤ 0.02	≤ 0.02

*Initial residual was 1.94 mg/L as Cl_2

Table 23: Free chlorine residual in samples collected from epoxy-coated pipe specimens exposed to chlorinated pH 8 extraction water

Pipe No.		Holding Time, h	Free Chlorine Residual (mg/L as Cl_2)	
LSLs	CSLs		LSLs	CSLs
Pb03	Cu03	6	0.17	0.33
Pb06	Cu06	24	0.08	0.12
Pb08	Cu08	96	≤ 0.02	≤ 0.02
Pb06	Cu06	240	≤ 0.02	≤ 0.02

*Initial residual was 2.00 mg/L as Cl_2

Table 24: Combined chlorine residual in samples collected from pipe specimens (both epoxy-coated and uncoated controls) exposed to chloraminated pH 8 extraction water

Pipe No.		Holding Time, h	Combined Chlorine Residual (mg/L as Cl_2)			Notes
LSLs	CSLs		LSLs	CSLs	Extraction Water*	
Pb04	Cu10	6	1.54	0.48	2.05	Uncoated control pipe specimens
Pb08	Cu08	6	0.27	0.83	2.05	Previously exposed to chlorinated extraction water
Pb01	Cu01	6	0.67	0.30	2.05	Never exposed to chlorinated extraction water
Pb07	Cu07	24	0.06	0.06	1.82	Never exposed to chlorinated extraction water
Pb01	Cu01	96	0.05	0.06	1.62	Previously exposed to chlorinated extraction water

*Initial residual was 1.94 mg/L as Cl_2

APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 25: Free chlorine residual in samples collected from epoxy-coated pipe specimens exposed to sequential 1-hour fill-and-dump tests using chlorinated pH 8 extraction water

	Free Chlorine Residual, mg/L as Cl_2		
Round	Pb06	Cu06	Extraction Water*
1	1.14	1.09	
2	1.32	1.24	
3	1.34	1.32	
4	1.39	1.34	1.98
5	1.38	1.35	
6	1.51	1.45	1.99
7	1.58	1.52	
8	1.55	1.48	
9	1.51	1.53	
10	1.56	1.50	1.96
11	1.77	1.76	
12	1.80	1.82	
13	1.79	1.80	
14	1.77	1.80	
15	1.80	1.78	1.96

*Initial residual was 1.99 mg/L as Cl_2

Table 26: Free chlorine residual in samples collected from epoxy-coated pipe specimens exposed to sequential 3-hour fill-and-dump tests using chlorinated pH 8 extraction water

	Free Chlorine Residual, mg/L as Cl_2			
	Pipe Specimen		Extraction Water	
Round	Pb06	Cu06	Initial	Final
1	73	76	99	98
2	83	86	96	99
3	87	88	101	99
4	87	89	98	99
5	89	90	99	99

APPENDIX D – EPOXY-COATED PIPE SPECIMEN DATA

Table 27: TOC concentration increases in samples collected from fill-and-dump experiments on epoxy-coated pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		TOC, mg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 1 (FD-01)	Dechlorinated Tap Water, pH 8		Extraction Water		3.53	3.53
		6	Pb04, Control	Cu10, Control	0.09	0.09
		6	Pb01	Cu01	-0.25	-0.07
		6	Pb02	Cu02	-0.17	1.25†
		24	Pb05	Cu05	0.12	-0.05
		96	Pb07	Cu07	0.23	0.46
		240	Pb05	Cu05	-0.35	-0.34
	Chlorinated Extraction Water, pH 8		Extraction Water		0.80/0.45*	
		6	Pb03	Cu03	0.16	1.36
		24	Pb06	Cu06	0.12	0.30
		96	Pb08	Cu08	12.5†,**	1.27
		240	Pb06	Cu06	0.32	0.66
	pH 6.5 Extraction Water		Extraction Water		0.35/0.29*	
		6	Pb04, Control	Cu10, Control	0.15	-0.04
		168	Pb04, Control	Cu10, Control	0.71	-0.01
		6	Pb01	Cu01	0.17	1.03**
		6	Pb02	Cu02	0.38	1.13
		168	Pb01	Cu01	0.49**	2.77**
		168	Pb02	Cu02	1.39	4.07†,**
		6	Pb03	Cu03	0.04	0.51
		168	Pb03	Cu03	0.20	1.42
Seven-Month Wait, then Fill-and-Dump Experiment 2 (FD-02)	Chlorinated pH 8 Extraction Water		Extraction Water		0.20/0.19*	
		6	Pb04, Control	Cu10, Control	1.67	0.21
		168	Pb04, Control	Cu10, Control	0.83	0.06
		6	Pb08	Cu08	1.26	0.71
		24	Pb02	Cu02	0.16	0.14
		24	Pb05	Cu05	0.61	0.81
		24	Pb09	Cu09	0.75	2.46
		168	Pb02	Cu02	0.32	0.35
		168	Pb05	Cu05	0.39	0.61
		168	Pb09	Cu09	0.69	1.27
	Re-Flushed, then Chlorinated pH 8 Extraction Water		Extraction Water		0.51**/0.17*	
		6	Pb02	Cu02		
		6	Pb05	Cu05		
		6	Pb08	Cu08		
		6	Pb09	Cu09	-0.02**	-0.08**
		24	Pb02	Cu02		
		24	Pb05	Cu05		
		24	Pb08	Cu08		
		24	Pb09	Cu09	-0.05	-0.05
		168	Pb02	Cu02	0.27	0.26
		168	Pb05	Cu05	0.31	0.35
		168	Pb08	Cu08	1.58	0.56
		168	Pb09	Cu09	0.52	0.54

*The data points represent extraction water used for a detention time of 6 – 24 hours and 7 days, respectively
†Statistical outlier (Grubb's, $\alpha = 0.05$), omitted from further analyses
**%RSD criteria was exceeded

APPENDIX E – PET-LINED PIPE SPECIMEN DATA

Table 28: Fill-and-dump testing matrix using PET-Lined pipe specimens, listed in order of test

Experiment	Extraction Water	Holding Time, h	Pipe No.		Notes
			LSLs	CSLs	
Fill-and-Dump Experiment 3 (FD-03)	Dechlorinated Tap Water, pH 8	6	Pb11	Cu11	Unlined Control
		6	Pb12	Cu12	
		6	Pb13	Cu13	
		24	Pb15	Cu15	
		96	Pb17	Cu17	
	Chlorinated pH 8 Extraction Water	6	Pb14	Cu14	
		24	Pb16	Cu16	
		96	Pb18	Cu18	
	pH 6.5 Extraction Water	6	Pb11	Cu11	Unlined Control
		96	Pb11	Cu11	Unlined Control
		6	Pb12	Cu12	
		6	Pb13	Cu13	
		96	Pb12	Cu12	
		96	Pb13	Cu13	
		6	Pb14	Cu14	
		96	Pb14	Cu14	
Stainless Steel Nipples Removed, Liners Cleaned, then Fill-and-Dump Experiment 4 (FD-04)	pH 6.5 Extraction Water	6	Pb11	Cu11	Unlined Control
		96	Pb11	Cu11	Unlined Control
		6	Pb19	Cu19	
		96	Pb19	Cu19	
		6	Pb12	Cu12	
		6	Pb13	Cu13	
		96	Pb12	Cu12	
		96	Pb13	Cu13	
		6	Pb14	Cu14	
		96	Pb14	Cu14	

APPENDIX E – PET-LINED PIPE SPECIMEN DATA

Table 29: Antimony concentration increases in samples collected from fill-and-dump experiments on PET-lined pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		Sb, µg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 3 (FD-03)	Dechlorinated Tap Water, pH 8	0	Extraction Water		0.19	
		6	Pb11, Control	Cu11, Control	3.94	0.19
		6	Pb12	Cu12	0.05	0.12
		6	Pb13	Cu13	0.05	0.09
		24	Pb15	Cu15	0.06	0.06
		96	Pb17	Cu17	0.15	0.15
	Chlorinated pH 8 Extraction Water	0	Extraction Water		≤ 0.06	
		6	Pb14	Cu14	≤ 0.06	0.08
		24	Pb16	Cu16	≤ 0.06	0.13
		96	Pb18	Cu18	0.09	0.18
	pH 6.5 Extraction Water	0	Extraction Water		≤ 0.06	
		6	Pb11, Control	Cu11, Control	1.32	≤ 0.06
		96	Pb11, Control	Cu11, Control	0.42	0.13
		6	Pb12	Cu12	0.14	0.13
		6	Pb13	Cu13	0.14	0.12
		96	Pb12	Cu12	0.40	0.44
		96	Pb13	Cu13	0.46	0.43
		6	Pb14	Cu14	0.09	0.09
		96	Pb14	Cu14	0.43	0.55
SS Nipples Removed, Liners Cleaned, Then Fill-and-Dump Experiment 4 (FD-04)	pH 6.5 Extraction Water	0	Extraction Water		Not Analyzed	Not Analyzed
		6	Pb11, Control	Cu11, Control		
		96	Pb11, Control	Cu11, Control		
		6	Pb19	Cu19		
		96	Pb19	Cu19		
		6	Pb12	Cu12		
		6	Pb13	Cu13		
		96	Pb12	Cu12		
		96	Pb13	Cu13		
		6	Pb14	Cu14		
		96	Pb14	Cu14		

APPENDIX E – PET-LINED PIPE SPECIMEN DATA

Table 30: Free chlorine residual in samples collected from PET-lined pipe specimens exposed to chlorinated pH 8 extraction water

Pipe No.		Holding Time, h	Free Chlorine Residual (mg/L as Cl_2)		
LSLs	CSLs		LSLs	CSLs	Extraction Water*
Pb14	Cu14	6	1.54	1.72	1.98
Pb16	Cu16	24	0.99	1.45	1.97
Pb18	Cu18	96	0.82	1.15	1.95

*Initial chlorine residual was 1.97 mg/L as Cl_2

Table 31: Free chlorine residual in samples collected from PET-lined pipe specimens exposed to sequential 24-hour fill-and-dump tests using chlorinated pH 8 extraction water

	Free Chlorine Residual, mg/L as Cl_2				
	Lined Pipe Specimen		Unexpanded PET liner	Extraction Water	
Round	Pb14	Cu14		Initial	Final
1	1.45	1.69	*	2.03	1.94
2	1.86	1.96	1.82	2.01	1.92
3	1.90	1.94	1.86	1.97	1.97

*Unexpanded PET Liner not tested until round 2

APPENDIX E – PET-LINED PIPE SPECIMEN DATA

Table 32: TOC concentration increases in samples collected from fill-and-dump experiments on PET-lined pipe specimens

Experiment	Extraction Water	Holding Time, h	Pipe No.		TOC, mg/L	
			LSLs	CSLs	LSLs	CSLs
Fill-and-Dump Experiment 3 (FD-03)	Dechlorinated Tap Water, pH 8	0	Extraction Water		3.34	
		6	Pb11, Control	Cu11, Control	0.00	-0.13
		6	Pb12	Cu12	0.01	0.02
		6	Pb13	Cu13	0.02	0.00
		24	Pb15	Cu15	0.11	0.02
		96	Pb17	Cu17	0.03	0.04
	Chlorinated pH 8 Extraction Water	0	Extraction Water		0.15	
		6	Pb14	Cu14	0.07	0.04
		24	Pb16	Cu16	0.21	0.12
		96	Pb18	Cu18	0.24	0.20
	pH 6.5 Extraction Water	0	Extraction Water		≤ 0.10/≤ 0.10	
		6	Pb11, Control	Cu11, Control	0.30	0.08
		96	Pb11, Control	Cu11, Control	1.02	0.11
		6	Pb12	Cu12	0.00	0.00
		6	Pb13	Cu13	0.06	0.00
		96	Pb12	Cu12	0.06	0.00
		96	Pb13	Cu13	0.00	0.00
		6	Pb14	Cu14	0.00	0.00
		96	Pb14	Cu14	0.00	0.00
Stainless Steel Nipples Removed, Liners Cleaned, Then Fill-and-Dump Experiment 4 (FD-04)	pH 6.5 Extraction Water	0	Extraction Water		Not Analyzed	Not Analyzed
		6	Pb11, Control	Cu11, Control		
		96	Pb11, Control	Cu11, Control		
		6	Pb19	Cu19		
		96	Pb19	Cu19		
		6	Pb12	Cu12		
		6	Pb13	Cu13		
		96	Pb12	Cu12		
		96	Pb13	Cu13		
		6	Pb14	Cu14		
		96	Pb14	Cu14		

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ABBREVIATION LIST

AL	= Action Limit
ANSI	= American National Standards Institute
BLL	= Blood Lead Level
cm	= Centimeter
CSL	= Copper Service Line
Cu##	= Copper Service Line Pipe Specimen Identification Number
DBPs	= Disinfection By-Products
EDCs	= Endocrine Disrupting Compounds
EPA	= United States Environmental Protection Agency
GFAAS	= Graphite Furnace Atomic Absorbance Spectrometry
HDPE	= High Density Polyethylene
ICP-MS	= Inductively Coupled Plasma Mass Spectrometry
IQ	= Intelligence Quotient
kg	= Kilogram
L	= Length
LCR	= Lead and Copper Rule
LSL	= Lead Service Line
M	= Molar
MCL	= Maximum Contaminant Level
MDL	= Method Detection Level
MDPE	= Medium Density Polyethylene
mL	= Milliliter
MSDS	= Material Safety Data Sheet
N	= Normality
NSF	= NSF International (formerly the National Sanitation Foundation)
OCCT	= Optimized Corrosion Control Treatment
PAC	= Project Advisory Committee
Pb##	= Lead Service Line Pipe Specimen Identification Number
PET	= Poly(ethylene terephthalate)
PTFE	= Polytetrafluoroethylene
PVC	= Polyvinylchloride
QA/QC	= Quality Assurance / Quality Control
SA	= Surface Area
SS	= Stainless Steel
STAR	= Science To Achieve Results
TETA	= Triethylenetetramine
TOC	= Total Organic Carbon
USB	= Universal Serial Bus
V	= Volume
W	= Watt
Ω	= Ohm

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